

ANIMAL FATS AND OILS

THEIR PRACTICAL PRODUCTION, PROPERTIES,
FALSIFICATION AND EXAMINATION

BY

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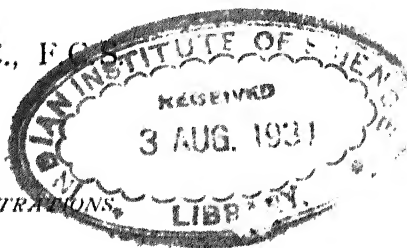
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REVISER'S PREFACE.

ALTHOUGH the animal oils and fats are essentially similar to vegetable oils and fats in composition, the raw materials require somewhat different methods of treatment for their extraction, hence it appeared to the author that a book treating specially upon animal fats would form a useful companion to those already published on "Vegetable Fats and Oils" and "Drying

The extent of the various industries connected with the preparation of margarine, lard, tallow, whale, and fish oils, dégras, wool fat, etc., proves that this is a most important one and that it must therefore interest a very large number of people. It should

be the object of a textbook of this kind to blend practical experience with scientific knowledge in such a way as to render it acceptable to the largest number of persons to whom it appeals, and the author is hopeful that he has been able to do so, at any rate to at least a fair extent, considering the large amount of material to be dealt with and the need to condense it within a moderate compass.

The third revised edition now issued has been consequently extended so as to include much new material, and at the same time the text has been thoroughly revised throughout up to date, it is therefore hoped that the book will prove useful to all those who are interested in the production and use of Animal Oils and Fats.



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ANIMAL FATS AND OILS.

CHAPTER I.

INTRODUCTION.

THE products known under the name of "animal fats" are closely related, both in their chemical and physical properties, to the vegetable fats. Like the latter, they are, for the most part, compounds of one or more fatty acids with glycerine. At ordinary temperatures, they are either solid, semi-fluid or perfectly liquid; and leave behind permanent greasy marks on paper. They dissolve slightly in boiling alcohol and completely in ether, benzene, and carbon bisulphide; can be mixed together when in a melted condition, *i.e.*, when heated, and are all lighter than water, so that they float on that liquid. When rubbed in thin layers on other substances, such, for example, as the skin, wood, etc., they repel watery liquids, and thereby afford a certain amount of protection against the penetration of such. Finally, they exhibit what is generally denoted as a "greasy feel" when handled.

The fats are encountered throughout the animal kingdom, in all its classes and subdivisions. They are met with in mammals, birds, amphibia, fish, and even in insects, and occur particularly in separate layers under the skin, interspersed in the flesh, between the intestines, or stored in the brain; a great part of the nutriment absorbed into the animal economy being converted into fat which under normal conditions is stored as a reserve material. Thus, under certain circumstances the fat accumulated in the animal body can serve to

support the individual, for a short time, as a means for the continuance of life during periods when the supply of nutriment is either reduced or entirely suspended. This is observed in the case of animals (*e.g.*, bats, bears, hedgehogs, etc.) which hibernate or lie dormant through the winter, awaking in spring, or in a favourable season, reduced in flesh.

The amount of fat stored up in the animal body is a particularly variable quantity, depending on the supply of food, mode of life and other circumstances. As a rule, the accumulation of fat is favoured by a secure and undisturbed existence, but may also be considerably increased by artificial means, as noted later. The different land animals useful on account of their fat are comparatively few in number, and comprise in fact only those that are bred on a large scale to be utilised partly for food and partly for technical purposes. Moreover, the fat of many animals is uneatable, being of unpleasant odour and flavour; and in the case of many others it is present in insufficient quantity to serve any industrial purpose.

There can be no reason to doubt that the fat of animals, equally with their flesh, was employed by man, even in the earliest times, for manifold purposes, although originally it was used for food alone. The use of fat for curative purposes—value in which respect is still attributed to bear's grease, badger's fat and dog's fat by country folk—came later. Fats for burning and the purpose of illumination, their application to the body to enable it to better withstand inclement weather, also for impregnating clothing and other articles in order to make them soft, supple and waterproof, probably formed the next stage of extension; finally, in very recent times, their technical utilisation has been considerably developed for soap-making, candles, etc.

As regards curative powers, the only fats at present used in any quantity are lard and cod-liver oil—very few at present believe that bear's grease and other fats have any healing

powers, the substance now sold under this name, it may be mentioned, being merely a mixture of lard and tallow.

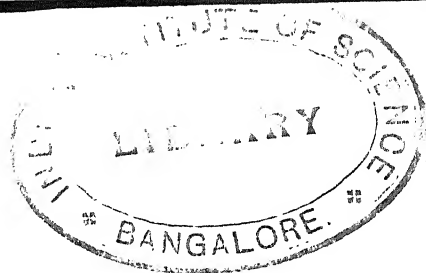
As progress was made in chemico-technical matters, and as the population increased, attention was naturally directed towards the recovery and utilisation of fats, we can also realise by the enormous consumption of soap, candles, etc., the great importance attaching to the production of animal fats in the present age. Moreover, the by-products obtained in the working up of fat play an important rôle, this being particularly the case with glycerine, which is now produced in enormous quantities. Whereas half a century ago the glycerine formed during saponification which passed into the spent lye and was simply allowed to run to waste along with the lye, is to-day a highly important article of commerce, the amount annually produced throughout the globe being now very considerable. Both medicinal glycerine of high grade, and several lower qualities for industrial use being made on a very large scale.

The preparation of fish oils has also greatly increased in extent, and many new sources of supply have been discovered. The best qualities of cod-liver oil are used for medicinal purposes, whilst the inferior grades are chiefly used for technical purposes, serving as emollients in leather dressing. Whale oil and menhaden oil are also now very largely used.

So far as the method of preparing animal fats is concerned, this was until comparatively recently of a very primitive description: the crude fat being melted or "rendered" in open pans heated by direct fire, either with or without water, perhaps melted again for purification, and afterwards placed on the market. The unpleasant exhalations attendant on some fat-rendering operations, especially when old fats (partly intermixed with putrefying flesh), bones, etc., were being treated, and which contaminated the atmosphere of the whole neighbourhood round such tallow-boiling establishments, led to the authorities insisting on a modification of the arrangements,

so that at the present time fat-melting works with their perfected appliances carry on their occupation without evolving any smell and without inconvenience to anyone in the vicinity. In recovering fat from bones, glue as well as fat is produced, the raw material being thereby fully utilised in a rational manner.

The importance pertaining to animal fats in the world's commerce can be estimated to some extent by the list of imports and exports of various countries, but these do not give an adequate conception of the magnitude of the animal fat industries, as they take no account of the enormous amounts which are locally consumed and for which in many cases there are no published data.



CHAPTER II.

OCCURRENCE, ORIGIN, PROPERTIES AND CHEMICAL CONSTITUTION OF ANIMAL FATS.

FAT is found in all the organs constituting the animal body—in individual places accumulated in large quantity—as well as in all animal fluids, with the exception of urine. Certain animals exhibit a greater tendency to secrete fat than others, the domestic animals having this faculty particularly well developed. In the animal organism fat is generally found enclosed in special cells, in larger amount in connective tissue, in the *panniculus adiposus* under the skin, in the plexus of the abdominal cavity, in the vicinity of the kidneys, in the marrow of the bones and spine, in the brain, in the liver and in the milk; and occurring pathologically in so-called fatty tumours and in fatty degeneration of the various tissues caused by disease.

Concerning the origin of the fat in the animal body the following may be asserted: The fat stored up in the bodies of animals fed on a generous diet does not consist solely of ready-formed fat absorbed from the food, but is to a considerable extent elaborated in the body from other chemical compounds. From an exhaustive study of the composition of the nutriment of the herbivorous animals, coupled with a knowledge of the remarkable changes which organic substances outside the organism may undergo, and reflection on the importance of the individual constituents of nutrition, Liebig came to the conclusion that the carbohydrates (starch, dextrin, sugar) played an important part in the formation of fat within

the body; and on the basis of his assumptions the opinion prevailed during several decades that the formation of fat from carbohydrates was an unassailable fact. In proof thereof were specially advanced the facts that in the carnivora, which, apart from fat, consume no non-nitrogenous food, the elaboration of fat is generally deficient but increases considerably when they are placed on a mixed diet with an excess of carbohydrates; that the bulk of the food of herbivorous animals consists of carbohydrates; and finally, that bees even when fed for a long time solely on wax-free honey or sugar are still able to produce wax—*i.e.*, a fatty body—without loss of health or weight.

The researches made by Voigt and Pettenkofer, however, showed that it was more probable that the albuminoid substances in the food are, apart from the ready-formed fats therein, the chief source of fat, and that quite a different interpretation must be placed on the functions discharged by the carbohydrates in this connection; the latter do not represent the special material from which the fat stored in the body is produced direct, but they are none the less essential (in the dietary of the herbivorous animals at least) in order that fat may be elaborated.

Pettenkofer and Voigt have demonstrated that in both carnivorous and herbivorous animals the decomposition of albuminoid substances invariably results in the separation of fat which in the course of subsequent alterations is either completely consumed or, when protected from further oxidation by the presence of other easily oxidisable substances as the carbohydrates, remains as a residue in the body and is laid up therein as a valuable store of reserve force ready to be drawn upon in time of need. The importance of the carbohydrates in the formation of fat is therefore restricted to inhibiting the combustion of the fat separated in the decomposition of the albuminoids, so that the fat may have the opportunity of accumulating within the tissues. Actually, the fat and

albuminoid matters of the dietary are always sufficient, even in the case of the enormous fat production exhibited by milch kine, to yield the fat required, fattening with carbohydrates being only efficient provided albuminoids are simultaneously supplied.

By means of a methodic system of dieting ("fattening" or "feeding") an increase of the fat and flesh of animals destined for the slaughter-house can be produced. As the body fills up in the course of fattening the animal assumes a condition of imperfect health, for which reason highly justifiable objections have been raised against over-fattening (a course of feeding first practised in England), not only from a veterinary standpoint but also in view of the utilisation of the meat. The most nutritious and best flavoured meat is obtainable only from animals in a condition ranging from incipient fattening up to the half-fat stage, whilst very fat beasts, on the other hand, yield chiefly tallow and fat, their flesh and blood being deficient in those constituents which most effectively act in the elaboration of matter in the animal economy.

The following animals are those whose fat is most prized and utilised: oxen, sheep, pigs and horses; among birds the goose almost exclusively; also the large marine mammals, such as the whale and seal, the dolphin, also among fishes the shark, and a number of smaller fish, such as the cod, ray, herring, sprat, sardine, anchovy, etc.

The positions in the animal body where the fat is stored are various. It is often encountered in considerable masses directly under the skin (as in the pig), or between the intestines (belly fat), in the brain (in the sperm whale), in the liver (of numerous fish), and, finally, distributed throughout the whole body, so that in order to recover it the entire carcase must be boiled or pressed (small fish, such as the anchovy, etc.).

At ordinary temperatures the animal fats when in a pure state are solid or liquid; the colour ranges from white (tallow, lard) to yellowish-white, pale yellow or brown (fish oils). The solid fats melt at between 20° and 45° C., and the liquid fats become semi-fluid or quite firm at temperatures from 5° C. downwards. The so-called boiling-points are various, and when heated still further all the fats are decomposed, their glycerine being converted into acrolein, a substance with a most unpleasant smell. The specific gravity of the animal fats is less than that of water, and can, as in the case of the vegetable fats, be regarded as a characteristic indication of their purity.

When fresh, the animal fats, for the most part, have an agreeable odour, the only exceptions being sundry fish oils; on the other hand, when old, they have generally a rancid and unpleasant, sometimes putrescent and even repulsive, smell (old bone fat, blubber, etc.). The flavour of some is agreeable, as in those generally employed for alimental purposes (butter and lard); in others (tallow, especially mutton tallow), unpleasant; and in the case of fish oils, occasionally nauseous. Nearly all fats will produce, even at the ordinary temperature, grease spots which do not disappear on warming, and even those of highest melting-point give rise to grease spots when heated. When absorbed by a wick, all fats will burn with a more or less bright, smoky and strong-smelling flame.

The solid fats when viewed under the microscope at the ordinary temperature appear throughout as a mixture of solid and liquid substances. The solid portion consists mainly of crystals in the shape of plates, needles or tufts, which have a composition different to the matrix. If the fat be warmed on a glass slip just to the melting-point, sundry solid amorphous granules will still generally be observable in the mass.

On cooling, the constituents crystallise out again, mostly in

the form of needles. In fats poor in olein the liquid portion appears in the form of drops, but it forms the fluid matrix in those rich in olein. This fluid mass when viewed under the microscope frequently appears not to be homogeneous, but to contain drops with a different power of refraction from the remainder.

The fats are insoluble (or, according to recent statements, soluble, though with extreme difficulty) in water, and but little soluble in cold alcohol, though generally slightly soluble in hot alcohol, and easily soluble in ether, benzene, petroleum ether, etc.

TRIGLYCERIDES.

Owing to the fact that mixed glycerides are usually present in fats it is extremely difficult to prepare pure triglycerides from them by processes of recrystallisation or in other ways. Thus, for instance, the deposit from an ethereal solution of lard after once crystallising has a melting-point of 62.0°C ., which is increased to 65.8°C . after six crystallisations from the same liquid, while beef tallow after once crystallising has a melting-point of 55.5°C ., which rises to 60°C . after six crystallisations.¹ As the melting-point of pure tristearin is $71^{\circ}\text{--}72^{\circ}\text{C}$. it is evident that none of these products consist of pure tristearin, but that they contain tristearin and tripalmitin in molecular combination.

Pure triglycerides are, therefore, usually prepared synthetically by heating the fatty acids with excess of glycerine in a sealed tube under pressure. The following triglycerides which occur in animal oils and fats have thus been prepared:—

Tributyryl, $\text{C}_8\text{H}_5(\text{O} \cdot \text{C}_4\text{H}_7\text{O})_3$. This is a colourless liquid which does not solidify except at extremely low temperatures. It distils unchanged at $182^{\circ}\text{--}184^{\circ}\text{C}$. under a pressure of 24 mm.,

¹ Kreis and Hafner.

has a density of 1.0324, and a refractive index of 1.48587 at $\frac{20^{\circ}}{4}$ C. (Scheij). Contained in butter fat.

Trivalerin, $C_3H_5(O \cdot C_5H_9O)_3$. Stated to be present in dolphin and porpoise oils.

Tricaproin, $C_3H_5(O \cdot C_6H_{11}O)_3$. Forms a colourless and tasteless liquid, which solidifies at about -60° C., and has a sp. gr. of 0.9817, and the refractive index 1.44265 at $\frac{20^{\circ}}{4}$ C. (Scheij). Stated to be present in butter fat, also present in coco-nut and palm-nut oils.

Tricaprylin, $C_3H_5(O \cdot C_8H_{15}O)_3$. Colourless and tasteless liquid solidifying at -15° C., and having the sp. gr. 0.9540, and the refractive index 1.44817 at $\frac{20^{\circ}}{4}$ C. (Scheij). This glyceride is also present in butter, coco-nut and palm-nut oils.

Tricaprin, $C_3H_5(O \cdot C_8H_{15}O_3)$. This glyceride is solid and crystalline, and melts at 31.1° C. Its density at $\frac{40^{\circ}}{4}$ C. is 0.9205, and refractive index at the same temperature 1.44461. It is present in butter fat, coco-nut and palm-nut oils.

Trimyristin, $C_3H_5(O \cdot C_{14}H_{27}O_3)$. Solid crystalline fat, melting at 55° C. Its density at $\frac{60^{\circ}}{4}$ C. being 0.8848, and its refractive index 1.44285. Trimyristin is contained in butter fat, lard, and human fat, but it is the characteristic glyceride of nutmeg butter.

Tripalmitin, $C_3H_5(O \cdot C_{16}H_{31}O)_3$. This is one of the most widely distributed of the triglycerides, being present in most oils and fats in association with triolein and tristearin. It is a lustrous white crystalline product, melting-point 65° C., sp. gr. at $\frac{80^{\circ}}{4}$ C. 0.8657, and refractive index 1.43807. Tripalmitin has, when quickly cooled, two apparent melting-points, one occurring at 45° - 46° C., and the other at 65° C.

Tristearin, $C_3H_5(O \cdot C_{18}H_{35}O)_3$. Glistening white crystalline product melting at 71° - 72° C., having a density at $\frac{80^{\circ}}{4^{\circ}}$ C. of 0.8621, and a refractive index at the same temperature of 1.43987 (Scheij).

Triarachin, $C_3H_5(O \cdot C_{20}H_{39}O)_3$. Contained in small amount in butter fat.

Triolein, $C_3H_5(O \cdot C_{18}H_{33}O)_3$. Forms an almost colourless oily liquid solidifying at -4° C. Its sp. gr. at 15° C. is 0.900.

Like their vegetable congeners, the animal fats consist principally of neutral glycerides of the fatty acids, and therefore have the general formula $C_3H_5(O \cdot OC R^1)$, wherein R^1 represents a monovalent hydrocarbon radicle. In addition to this the fats may also contain free fatty acids. Sperm oil and the spermaceti obtained therefrom contain, however, no glycerides, but ethers of the higher alcohols of the fatty series, and should (along with shark oil and probably all the oils derived from marine animals and having a sp. gr. of less than 0.888 at 15° C.) properly be relegated to the category of the waxes.

ANIMAL WAXES.

Cetyl Palmitate or Cetin, $C_{16}H_{33} \cdot O \cdot CO \cdot C_{15}H_{31}$, is the principal constituent in spermaceti, from which it can be prepared by fractional precipitation from ether. It forms colourless crystals melting at 55° C.

Ceryl Cerotate, $C_{26}H_{53} \cdot O \cdot CO \cdot C_{25}H_{51}$. This is a constituent of Chinese insect wax, but it is also said to occur in wool fat. White glistening scales melting at 82.5° C.

All the animal fats may be saponified, *i.e.*, decomposed into fatty acids and glycerine, by alkalis, dilute acids and by means of superheated steam. By this process the following acids and alcohols have been separated from the fats and waxes:—

A. ACIDS.

1. *Saturated Acids of the General Formula $C_nH_{2n}O_2$, of the Acetic Series :—*

Butyric acid . . .	$C_4H_8O_2$
Isovaleric acid . . .	$C_5H_{10}O_2$
Caproic acid . . .	$C_6H_{12}O_2$
Caprylic acid . . .	$C_8H_{16}O_2$
Capric acid . . .	$C_{10}H_{20}O_2$
Lauric acid . . .	$C_{12}H_{24}O_2$
Myristic acid . . .	$C_{14}H_{28}O_2$
Isocetic acid . . .	$C_{15}H_{30}O_2$
Palmitic acid . . .	$C_{16}H_{32}O_2$
Daturic (margaric) acid . . .	$C_{17}H_{34}O_2$
Stearic acid . . .	$C_{18}H_{36}O_2$
Arachidic acid . . .	$C_{20}H_{40}O_2$
Behenic acid . . .	$C_{22}H_{44}O_2$
Lignoceric (carnaubic) acid . . .	$C_{24}H_{48}O_2$
Cerotic acid . . .	$C_{26}H_{52}O_2$
Melissic acid . . .	$C_{30}H_{60}O_2$

2. *Unsaturated Acids (with Double Bond) of the General Formula $C_nH_{2n-2}O_2$ (Oleic Series) :—*

Tiglic acid . . .	$C_5H_8O_2$
Hypogæic acid } . . .	$C_{16}H_{30}O_2$
Phytetoleic acid {	
Oleic acid . . .	$C_{18}H_{34}O_2$
Doeglic acid . . .	$C_{19}H_{36}O_2$
Jecoleic acid . . .	$C_{19}H_{36}O_2$
Gadoleic acid . . .	$C_{20}H_{38}O_2$
Erucic acid . . .	$C_{22}H_{42}O_2$

3. *Acids (with Triple Bond) of the General Formula $C_nH_{2n-4}O_2$ (Linolic Series) :—*

Linolic acid . . .	$C_{18}H_{28}O_2$
Elæomargaric acid . . .	$C_{18}H_{32}O_2$

4. *Acids of the General Formula $C_nH_{2n-6}O_2$ (Linolenic Series) :—*

Linolenic acid . . .	$C_{18}H_{30}O_2$
Isolinolenic acid . . .	$C_{18}H_{30}O_2$

5. *Hydroxy Acids of the General Formula $C_nH_{2n-2}O_3$ (Ricinoleic Series) :—*

Ricinoleic acid . . .	$C_{18}H_{34}O_3$
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B. ALCOHOLS.

 1. *Trivalent Alcohol of the Formula* $C_nH_{2n+2}O_3$:—

 Glycerol (glycerine) . . . $C_3H_8O_3$

 2. *Monovalent Alcohols of the Fatty Series, with the Formula* $C_nH_{2n+2}O$:—

 Cetyl alcohol (ethal) . . . $C_{16}H_{34}O$

 Octodecyl alcohol . . . $C_{18}H_{38}O$

 Ceryl (iso-ceryl) alcohol . . . $C_{27}H_{56}O$

 Myricyl (melissyl) alcohol . . . $C_{30}H_{62}O$

 3. *Monovalent Aromatic Alcohols (Sterols)* :—

 Cholesterol . . . $C_{27}H_{46}O$

 Isocholesterol . . . $C_{27}H_{46}O$

 Phytosterol (Sitosterol) . . . $C_{27}H_{46}O$

The fats consist most frequently of the glyceride of palmitic acid (palmitin, tri-palmitin), $C_3H_5(C_{16}H_{31}O_2)_3$; the glyceride of stearic acid (stearin, tri-stearin), $C_3H_5(C_{18}H_{35}O_2)_3$; and the glyceride of oleic acid (olein, tri-olein), $C_3H_5(C_{18}H_{33}O_2)_3$. Other acids occur in small quantities in various fats, a high proportion of such an acid being generally characteristic of a certain fat. Such are :—

Butyric acid, as a glyceride (butyrin), in the proportion of 5 to 7 per cent. in cow's butter.

Isovaleric acid, occurring in combination with glycerine in dolphin and porpoise oils.

Of the *caproic acids*, isobutylic acid occurs as caproin in butter, in which caprylic and capric acids also are present in the form of caprylin and caprin; so that cow's butter contains about 8 per cent. of the glycerides of these acids.

Physetoleic acid, in sperm oil; and

Doeglic acid, in Arctic sperm oil.

The solid and liquid animal fats differ from one another in consistency, this being dependent on the varying proportions of the individual glycerides present in each; stearin and palmitin being solid at the ordinary temperature, whereas olein

is liquid. All rancid fats contain free fatty acids, the formation of which results from a decomposition effected by the air, whereby glycerine and free fatty acids are produced, but other changes are brought about by bacteria and by oxidation.

The saturated fatty acids up to capric acid, and the oleic, doeglic and various vegetable fatty acids are liquid at the ordinary temperature, the remainder being solid. Butyric, caproic, caprylic and capric acids, being capable of distillation without decomposition, under ordinary pressure, are styled *volatile fatty acids*, and are also more soluble in water than the other fatty acids.

FATTY ACIDS.

Butyric Acid, $\text{HC}_4\text{H}_7\text{O}_2$. The acid is liquid at the ordinary temperature and exhibits, when freshly distilled, an odour resembling that of acetic acid, but in a dilute condition has a very strong smell like rancid butter. It boils at 162.3°C ., sets in a flaky condition at -19°C ., has a sp. gr. of 0.958 at 14°C ., and refractive index at 20°C . = 1.39906 (Scheij). It is readily soluble in water and can be separated therefrom as oily drops by the addition of calcium chloride or by common salt; with alcohol and ether it is miscible in all proportions.

Solutions of butyric acid have a corrosive acid taste, redden litmus and decolorise a slightly alkaline phenolphthalein solution. Methyl orange also is reddened by solutions of butyric acid containing no butyric salts. If a dilute aqueous solution of this acid be distilled the acid passes over completely in the distillate; in the case of very dilute solutions the attainment of this object is facilitated by previously neutralising the acid with soda, concentrating the liquid by evaporation, and distilling after adding dilute sulphuric acid.

Concentrated butyric acid when warmed with alcohol and concentrated sulphuric acid forms butyric-ethyl-ester (ethyl butyrate), which is recognisable in small quantities by its very

agreeable odour of pine-apple. To detect the presence of butyric acid in very dilute solutions by this reaction the liquid is neutralised with soda, evaporated to dryness, and the residue warmed with alcohol and sulphuric acid. A portion of the butyric acid is also etherised during the saponification of fats containing this acid, by strong alcohol and caustic potash.

Caproic Acid, $\text{HC}_6\text{H}_{11}\text{O}_2$. Of the isomeric caproic acids isobutylic acid occurs in animal fats to a slight extent. It is a liquid, soluble in coco-nut and palm-nut oils, but not miscible with water, and possesses a smell resembling that of perspiration. The sp. gr. is 0.925 at 20° C., refractive index at 20° C., 1.41635 (Scheij.); boiling-point, 199.7° C.; setting-point, below - 18° C., at which temperature it is still liquid.

Caprylic Acid, $\text{HC}_8\text{H}_{17}\text{O}_2$, is also liquid, but sets, on cooling to 12° C., to a crystalline mass, melting at 10.5° C., slightly soluble in water. Boiling-point, 230° to 237° C.; sp. gr. at 20° C., 0.914; refractive index at 20° C., 1.42825. It is present in butter fat and in coco-nut and palm-nut oils. It has an intense smell of perspiration and is soluble in 400 parts of water.

Capric Acid, $\text{HC}_{10}\text{H}_{19}\text{O}_2$, forms at the ordinary temperature fine flakes which melt at 31.3°-31.4° C. and emit a goaty odour. The sp. gr. of the melted acid is 0.94 at 37° C.; it boils at 268°-270° C., and 1 part of the acid is soluble in 1000 parts of boiling water. Is contained in butter fat, fat from goat's milk, and in coco-nut and palm-nut oils.

Palmitic Acid, $\text{HC}_{16}\text{H}_{31}\text{O}_2$. Pure palmitic acid can be obtained in the form of fine tufted groups of needles, or, after melting and re-solidification, as a crystalline scaly mass with a nacreous lustre; it is tasteless and inodorous, melts at 62° C., and at this temperature has in the liquid state a sp. gr. of 0.8527; refractive index at 60° C., 1.4324 (Ruttan). At about 350° C. it is for the most part volatile without decomposition.

and will boil at 268.5°C . when the pressure is reduced to 100 mm. of mercury.

When melted palmitic acid is placed on paper, or an alcoholic or ethereal solution of the acid is left to evaporate thereon, a grease spot is produced. This acid is soluble with difficulty in cold alcohol, 100 parts of the solvent taking up only 9.32 parts of acid, but it is very readily dissolved by boiling alcohol, and may, therefore, be very easily re-crystallised from this solvent. The alcoholic solution has an acid reaction.

Dilute acids have no action on palmitic acid, but it dissolves in concentrated sulphuric acid, re-crystallising, however, unchanged on dilution. Boiling concentrated nitric acid attacks it very slowly. The salts of palmitic acid are very similar to those of stearic acid, but are somewhat more readily soluble.

Stearic Acid, $\text{HC}_{18}\text{H}_{35}\text{O}_2$. Pure stearic acid crystallised from alcohol solution forms white lustrous laminæ which melt at 69.2°C . to a perfectly colourless fluid and set to a translucent crystalline mass on cooling. When heated to 360°C . it begins to boil and partly decomposes, but may be distilled without alteration under reduced pressure—at 291°C . under a pressure of 100 mm. When distilled by superheated steam it also passes over without apparent decomposition, but, according to observations made in the distillation of fatty acids on a working scale, a small portion is really decomposed, being converted into solid hydrocarbons of the $\text{C}_n\text{H}_{2n-2}$ series.

The sp. gr. of this acid at 11°C . is almost identical with that of water, but at higher temperatures it floats in the latter liquid owing to its greater coefficient of expansion. The sp. gr. of the acid melted at $\frac{69.2^{\circ}}{4^{\circ}}\text{C}$. is 0.8454.

Stearic acid is devoid of flavour or smell, feels greasy, and will leave a greasy mark on paper whether applied in the melted state or in solution. It is insoluble in water, but

readily soluble in hot alcohol, though in cold alcohol it is even more difficult to dissolve than palmitic acid, one part of stearic acid dissolving in 70 parts of absolute alcohol at 15°C .; ether dissolves it readily, and at 23°C . 1 part of benzene will dissolve 0.22 part, and 1 part of carbon bisulphide 0.3 part of stearic acid at 23°C .

Anchidic Acid, $\text{HC}_{21}\text{H}_{39}\text{O}_2$, is stated to be present in small amount in butter fat. It forms glistening crystalline scales, is readily soluble in hot alcohol but almost insoluble in cold alcohol, but soluble in ether, benzene, chloroform, and benzene. The melting-point of the acid is 77°C .

Cerotic Acid, $\text{HC}_{26}\text{H}_{50}\text{O}_2$. This acid is stated to occur as ceryl cerotate in wool fat; it is also the free acid present in beeswax. Melting-point 78°C . and density at $\frac{7}{4}^{\circ}\text{C}$. = 0.8359.

It crystallises from solution in alcohol in lustrous white scales or in fine needles. It is readily soluble in hot alcohol, but practically insoluble in this liquid in the cold.

Hypogæic Acid, $\text{HC}_{16}\text{H}_{29}\text{O}_2$. White needles, melting at 33°C ., gradually turning brown and decomposing in the air, forming liquid fatty acids of rancid flavour.

Phytotoleic Acid, $\text{HC}_{16}\text{H}_{29}\text{O}_2$, has the same composition as the preceding acid, but differs considerably therefrom in being unaltered by the action of nitrous acid. It melts at 30°C . It is found in sperm oil.

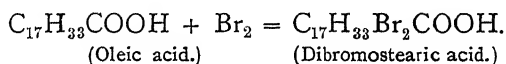
Oleic Acid, $\text{HC}_{18}\text{H}_{33}\text{O}_2$. Owing to the fact that oleic acid is usually accompanied by other unsaturated acids it is difficult to prepare it in a perfectly pure state. In this condition it appears as a colourless and inodorous oil which sets at 4°C ., but does not melt again below 14°C .; the sp. gr. at this temperature is 0.898; refractive index 1.4638 at 15°C . Under ordinary pressure the acid cannot be distilled without decomposition, but when heated by steam at a temperature of 250°C . it passes over unchanged.

When perfectly pure it does not redden litmus paper, but, on the other hand, it exerts an acid reaction on phenolphthalein, since the fresh fatty acid mixture (very rich in oleic acid) prepared from fats by saponification and subsequent acidification of the soap solution can be accurately titrated by means of this indicator.

When exposed to the air, oleic acid becomes yellowish or yellow, acquires a rancid odour and reddens litmus.

This acid is insoluble in water, but readily soluble even in dilute cold alcohol. It may, however, be separated from this solution by the addition of a large volume of water. An attempt has been made by David to found a method of separation for this acid on account of its greater solubility than the solid fatty acids in a mixture of alcohol, water and acetic acid.

If to 7 parts by weight (1 molecule) of oleic acid there is gradually added 4 parts of bromine, with continual shaking, the whole of the bromine will be absorbed and dibromostearic acid will be formed. From its method of preparation this substance is known as the dibromide of oleic acid. The reaction occurs according to the equation :—



When suitably purified the product forms a clear oil. A similar reaction in which iodine replaces bromine is obtained with an alcoholic solution of oleic acid in presence of an alcoholic solution of iodine containing mercuric chloride. The amount of iodine absorbed by oleic acid is 90 per cent. (Hubl's test).

If nitrous acid be allowed to react on oleic acid the latter sets hard in a short time and becomes converted into the isomeric elaidic acid, which may, by re-crystallisation from alcohol, be obtained in the form of plates melting at 45° C.

Doeglic Acid, $\text{HC}_{19}\text{H}_{35}\text{O}_2$, has received but little attention hitherto; it is a yellow oil similar to oleic acid and setting at 4°C . It is found in Arctic sperm oil.

Jecoleic Acid, $\text{HC}_{19}\text{H}_{35}\text{O}_2$. This acid is stated by Heyerdahl to be present in cod-liver oil.

Gadoleic Acid, $\text{HC}_{20}\text{H}_{37}\text{O}_2$. According to Bull, cod-liver oil, herring oil, and whale oils contain an acid which he calls gadoleic acid and to which the above formula is ascribed.

Linolic Acid, $\text{C}_{18}\text{H}_{32}\text{O}_2$. The characteristic acid of linseed oil is also present in appreciable quantities in some animal fats, *e.g.*, lard, hare fat, and horse fat. It is a colourless oily liquid which does not solidify at -18°C . and has a density at 18°C . of 0.9026. It is easily soluble in alcohol and ether and absorbs oxygen rapidly on exposure to the air. Linolic acid combines with 4 atoms of bromine to form the tetrabromide, $\text{C}_{18}\text{H}_{32}\text{O}_2\text{Br}_4$, which melts at 114°C .

ALCOHOLS.

Cetyl Alcohol, $\text{C}_{16}\text{H}_{34}\text{O}$, occurs in spermaceti in combination with palmitic acid as cetyl palmitate. It forms a colourless crystalline material, melts at 50°C . and boils without decomposition at 344°C . Its density at $\frac{60^\circ}{4^\circ} \text{C}$. is 0.8105, and it is insoluble in water but easily soluble in alcohol and ether.

Octodecyl Alcohol, $\text{C}_{18}\text{H}_{38}\text{O}$, also occurs as an ester in spermaceti. It crystallises from alcohol in glistening white scales melting at 59°C . At ordinary pressure it decomposes on heating but may be distilled under diminished pressure.

Glycerol or Glycerin, $\text{C}_3\text{H}_8\text{O}_3$. This alcohol is the basis of all true fats (glycerides) and is obtained from them by saponification with alkalis or hydrolysis with acids. When pure it forms a thick colourless transparent liquid, very sweet and hot to the taste but without odour. Its density at 15°C . is 1.26468, refractive index at 15°C . 1.4742, and boiling-point,

290° C. On distilling at this temperature under ordinary pressure, however, there is a slight amount of decomposition, but it distils unchanged under diminished pressure.

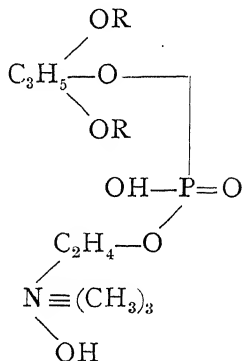
Cholesterol, $C_{27}H_{46}O$. This alcohol is present in large proportion in wool fat, while "lanolin" consists almost entirely of it. It is also contained in fat of egg yolks. Pure cholesterol crystallises in needles which melt at 148°-150° C. It is insoluble in water but soluble in alcohol, readily soluble in ether, chloroform, and carbon bisulphide.

Isocholesterol, $C_{27}H_{46}O$, is also present in wool fat. It forms fine needles which melt at 137°-138° C. Slightly soluble in cold alcohol, but more freely on heating, easily soluble in ether.

PHOSPHATIDES.

In different parts of the animal system and in plants there occur, usually in association with ordinary fats, certain peculiar fatty substances which contain both phosphorus and nitrogen, to which the name "phosphatides" was applied by Thudichum. These substances have a somewhat complicated constitution, but they are regarded as derived from glycerol, one hydrogen of which is replaced by phosphoric acid, and the other two by fatty acids (palmitic, stearic, oleic, etc.), while a choline radicle replaces one hydrogen atom of the phosphoric acid. Other bases besides choline appear in some cases to be present and there is usually present calcium and iron in small amounts. The phosphatides appear to play a very important part in living organisms, as they are almost universally present in living cells. They are specially abundant in the brain and in the ova of animals and in leguminous seeds.

Lecithin.—The best known of the phosphatides is lecithin, which is an optically-active substance and therefore has the assymetric formula :—



Lecithin is found in the following quantities in different parts of the body: brain, 16 per cent.; cardiac muscle, 12·5 per cent.; kidneys, 8·5 per cent.; liver, 4·3 per cent.; and nerve tissue, 2·5 per cent.; in yolk of egg it is present to the extent of 9 per cent.; in peas, 1·2 per cent.; and in beans 0·8 per cent. Lecithin is soluble in ether, etc., and therefore appears in the fat extracted by solvents. It is usually estimated by determining the amount of phosphorus in the extracted oils. Lecithin may be prepared from egg yolk fat, which contains about 22 per cent. of this substance, by heating it with alcohol and then filtering; the lecithin is contained in the filtrate and may be obtained by evaporation. It is also precipitated from a strong ethereal solution by adding acetone, in which it is insoluble. Neither of these products, however, are quite pure, although they are quite suitable and are used for commercial purposes. Pure lecithin is obtained by precipitating a solution with cadmium chloride as it forms an insoluble cadmium compound. It is prepared in a similar manner from vegetable sources for commercial purposes.

Lecithin is a reddish-brown or yellowish-brown transparent jelly without odour or taste. It shows a fibrous structure on pulling, and on heating it chars but does not melt. Commercial lecithins contain from 3·5-4·7 per cent. of phosphorus

and 1.9-2 per cent. of nitrogen, the ratio $\frac{P}{N}$ being about 1.8. The iodine value varies from 60 to 65.

The reviser has examined three samples of lecithin: (1) Ovo-lecithin, pharmaceutical, manufactured in France; (2) Lecithin extracted by self from hen's egg yolk fat by alcohol; (3) Lecithin extracted from duck's egg yolk fat by alcohol.

	1.	2.	3.
Iodine value (Wijs' method)	59.86	77.34	93.5
Phosphorus	3.58	3.53	2.91
Nitrogen	2.28	1.94	1.84
Ratio $\frac{P}{N}$	1.57	1.82	1.58

Lecithin is used as a medicine in nervous diseases and for anæmia, rickets, etc., also as a constituent of margarine (see p. 131).

Vitamines.—There are now known to be present in many kinds of food very small quantities of certain vital principles which are absolutely essential to nutrition and growth, these substances have been named "vitamines" by Dr. Fink, and as shown by Dr. F. G. Hopkins, Professor of Bio-chemistry at Cambridge, their absence quickly leads to malnutrition and paralysis. The constitution of these products is not yet known, as it has so far been impossible to obtain them in a pure state. They do not contain phosphorus. They are soluble in water and in 80 per cent. alcohol but not in absolute alcohol, and there appears to be at least three of these substances with different function. One of them is soluble in oils and is contained in egg yolk, cod-liver oil, butter and margarine from animal fats. Lard is the only animal fat which does not contain it, but it is not present in vegetable fats or oils and is therefore not to be found in margarine prepared exclusively from the latter. On this ground alone vegetable margarines must be regarded as less nutritive than those containing beef

oleo, although other foods partaken of at the same time may make up the deficiency.¹

HYDROCARBONS.

Saturated Hydrocarbons in Shark Oil.—In the *Journal of Industrial and Engineering Chemistry* Mitsumaru Tsujimoto gives some particulars of the oil from the liver of the basking shark (*Cetorhinus maximus*, Gunner), Japanese—"Uba-Zamé". This, it appears, is the largest of the sharks, and therefore the largest of the true fishes, the liver sometimes weighing as much as a ton and yielding some 5 cwts. of oil. Three samples of the oil from the livers were examined; these being pale yellow or orange yellow in colour, with a low gravity, and containing 41.92-55.51 per cent. of unsaponifiable matter. One of the samples gave the following constants:

Specific gravity at $\frac{15^{\circ}}{4}$ C., 0.8839; acid value, 1.09; saponification value, 102.45; iodine value (Wijs), 1.4773; butyro-refractometer at 20° C., 78.2; unsaponifiable matter, 41.92 per cent. The unsaponifiable matter was for the most part liquid, and on distillation in an oil bath 10 per cent. passed over at 170°-190° C. This had a refractive index of 1.4775 at 20° C., and differed from squalene (a highly unsaturated hydrocarbon, which had previously been discovered in other Japanese shark-liver oils). At 244°-260° C. a further 25 per cent. distilled over, consisting mainly of squalene ($C_{30}H_{50}$). The first distillate was subsequently purified by treatment with caustic soda and re-distilled in a partial vacuum yielding a liquid of sp. gr. 0.7868 at 15° C. and refractive index 1.4398 at 20° C. The iodine value was only 4.4, therefore the product is practically a saturated body. At 766 mm. pressure it distilled practically unchanged at a temperature of 296° C.,

¹ Prof. W. Ramsden, *Jour. Soc. Chem. Indt.* 1918, T., p. 53.

and was apparently a pure substance. An analysis of the oil gave carbon 84.39 and hydrogen 15.02 per cent., and the molecular weight determined by the freezing-point method mean of two experiments (265 and 252) was 259. These figures agree closely with the formula $C_{18}H_{35}$, $C_{17}H_{36}$ and $C_{19}H_{40}$ are also very close. If the formula $C_{18}H_{38}$ be adopted, then it is an octadecane, and as the nominal octadecane is solid, it follows, that this must be an iso-octadecane, which would account for the low boiling-point of the substance. It is stated that the hydrocarbons in this particular sample of oil are certainly not due to adulteration with petroleum or other products, as they differ materially in their properties from such, but although they were present in one sample they were absent from the other two samples examined.

Unsaturated Hydrocarbons in Shark-Liver Oil.—In the *Journal of Industrial and Engineering Chemistry* there also appears a very interesting paper by Mitsumaru Tsujimoto upon a highly unsaturated hydrocarbon which he has discovered in shark-liver oil. The author first refers to the fact that hydrocarbons are more frequently to be found in fatty oils than is generally believed. For instance, P. Matthes and O. Kohdeck separated a hydrocarbon having the composition $C_{30}H_{48}$ from cacao butter. The former chemist, along with H. Sander also obtained a hydrocarbon, $C_{20}H_{42}$, melting-point $69^{\circ} C.$, from laurel oil, which they named "laurane". Matthes and W. Heintz discovered a similar hydrocarbon, $C_{20}H_{42}$, melting-point $69^{\circ} C.$, in parsley-seed oil, to which the name "petrosilene" was given, while Power and Lees isolated from kōsam-seed oil another hydrocarbon, $C_{31}H_{64}$. In animal oils, especially those derived from insects, large quantities of hydrocarbons are said to occur; especially is this the case with chrysalis oil, from which Menozzi and Moreschi isolated two hydrocarbons, one of which, melting at $62\frac{1}{2}^{\circ} C.$, had the composition $C_{28}H_{58}$, while the other had a melting-point of $41^{\circ}-42^{\circ} C.$ Hydrocar-

bons have also been obtained from cantharides and Melalontha oils. The examination of what is termed "sterol free unsaponifiable matter" of oils appears to be of great value, and, according to the author, may possibly lead to the identification of oils when all other means fail. After removal of the sterols, which has been made possible since the valuable researches of Windaus, who used digitonin for the purpose, the residue may consist of hydrocarbons, alcohols, and ketones. J. Marcusson and G. Meyerheim, as also P. Berg and J. Augerhausen, have testified to the value of an investigation of the sterol free unsaponifiable matter thus prepared, and, in fact, the last two authors have founded upon its examination a method of distinguishing mowrah fat from shea butter, which could not be done by any other method.

Turning now to marine animal oils, the author states as a well-known fact that shark-liver oil contains a high percentage of unsaponifiable matter of which little is known, but which has been usually regarded as at any rate largely composed of cholesterol, although as a matter of fact a high percentage of hydrocarbon is really present. Allen states that in one sample of shark oil which he examined a large proportion of unsaponifiable matter resembling rosin oil was found, and this he regarded as an adulterant, a conclusion which the author believes was probably not correct. Ten years ago the author published an account of his researches upon the liver oil of a shark which was caught in the Sagami Sea, this being known as "kuroleozamé oil". The oil contained 56.13 per cent. of unsaponifiable light yellow oil, having the remarkably high iodine value of 381, and the composition of which is represented by the formula $C_{10}H_{18}$. Among the large number of species of shark which are known to exist only a few are caught because of their liver oil. Careful examination by the author tended to prove that liver oils having a gravity less than 0.900 contained more or less hydrocarbon, which increased in amount as the

gravity became lighter. As the oils known as "ai-zamé" and "heratsuno-zamé" were the lightest known, it was believed that these two would best repay examination, a conclusion which was amply verified. Ai-zamé oil is obtained from the liver of *Squalus mitsuleurii*, and contained 87 to 90 per cent. of unsaponifiable matter, 79 per cent. of which was found to consist of a hydrocarbon oil having the composition $C_{30}H_{50}$, half of which distilled over between 240° and 256° C. It had a sp. gr. at 15°_4 C. of 0.8587, iodine value 388 (Wijs), and refractive index at 20° C. of 1.4965. Heratsumo-zamé oil is derived from *Deama eglantina*, contains the same hydrocarbon as above, but also one or more other hydrocarbons of lower boiling-point, this fraction having a composition represented by the formula $C_{28}H_{38}$.

When fats or fatty acids are treated with caustic alkalis or lead oxide they are saponified. Concentrated alkaline lyes effect saponification with great difficulty, and therefore none but dilute lyes should be used. In the process of saponification stearin and palmitin are more quickly decomposed than olein.

On being melted with caustic potash, oleic acid is changed into palmitic acid; erucic acid into arachidic acid; hypogaic acid into myristic acid, and their isomers are decomposed into palmitic, arachidic, myristic and acetic acid. Concentrated sulphuric acid, in proportions of from 8 to 10 per cent., forms with fats at high temperatures sulpho acids, which, when treated with water, split up again into free fatty acids, glycerine and sulphuric acid. If fats or oils be treated with alkali carbonates, albumin, or an aqueous solution of gum-arabic, they are thereby emulsified, the same result being produced by the action of ammonia. Alcoholic ammonia solution when allowed to act for a long time at ordinary temperature converts the fats into acid amides.

Chlorine and bromine form *substitution products* with the fats or fatty acids of the methane series. Iodine does not act in this way but forms, as do also the above reagents, *addition products* with the glycerides or the fatty acids of the unsaturated hydrocarbons.

Nitric acid exerts a strong oxidising reaction on the fats, etc., forming oxalic acid, succinic acid and adipinic acid.

On exposure, especially in thin layers, to the air, the animal fats become rancid; in no instance, however, do they dry, as do certain of the vegetable oils, to a solid skin, but remain always in their original, more or less greasy, condition. In becoming rancid a part of the non-volatile fatty acids, especially oleic acid, is liberated, and occasionally a complete separation into fatty acids and glycerine occurs. Rancidity spreads in solid animal fats to a much smaller extent than in the vegetable oils, and they keep longer and better in proportion as their olein content is low and that of the glycerides of the solid fatty acids high. This condition (rancidity) is well known to be in a high degree prejudicial to the animal fats, on account of its effect on the flavour. Under defective storage conditions and under the influence of a favourable temperature, rancidity develops so quickly that butter, for example, may become uneatable in a few days if insufficiently protected from the air. The opinions prevailing on the causes of rancidity are somewhat divergent, but it cannot be doubted that when air is sufficiently excluded rancidity, if not altogether prevented, can be retarded for a considerable time.

Liebig was of opinion that rancidity is induced by the influence of the extraneous substances present in the fat; Löhwig assumed a fermentative action in presence of water and air; whereas Kosch considered it due to oxidation by atmospheric oxygen, an opinion also shared by Duclaux; and Berthelot ascribes it more particularly to moisture, the action of which is facilitated by the presence of extraneous substances,

oxidation being merely a concomitant reaction. Von Rechenberg, Flügge, Paschulin, H. Schulz and Neneki believe rancidity to be caused by the action of unorganised fat-destroying ferments, or by microbes. Gröger assumes that in becoming rancid the fat is decomposed by water into fatty acids and glycerine, followed by oxidation through the action of atmospheric oxygen, both on the fatty acids and on the glycerine, since the latter is no longer detectable in a free state. The fatty acids are split up into substances (acids), poorer in carbon but rich in oxygen, belonging partly to the fatty acid series and partly to the oxalic acid series. Among the latter class azelaic acid ($C_9H_{16}O_4$) has been identified.

Ritsert, who occupied himself very closely with the elucidation of the causes of rancidity, demonstrated that the rancidity of pure fat is not caused by either aerobic or anaerobic bacteria. The fat when protected from air and light remained perfectly sweet, and germs inoculated therein died, although able to live in rancid fat. Neither could fermentative action be assumed, since sterile fat heated in a closed vessel for several hours at 140° C. becomes rancid under the influence of light and air. Moisture is non-essential to rancidity, dry fat becoming more rancid under the influence of air than when moist; but the process is one of oxidation solely, induced by atmospheric oxygen, without the collaboration of any organisms. The operation proceeds more rapidly in proportion as light is admitted, oxygen not being absorbed when light is excluded, neither does air cause rancidity when deprived of its oxygen. Pure lard kept in the dark remains sweet even after a lapse of two months. The carbon dioxide of the air has the same influence on fat in the absence of light as pure carbonic acid, only less intense, and is absorbed by fat both in the light and in the dark in comparatively small amount, the fat becoming thereby tallowy, but not rancid. Both nitrogen and hydrogen are inactive towards fat, whether in

the light or in the dark, and pure butter behaves exactly like pure lard.

Aerobic and anaerobic bacteria can live on rancid fat, but not if an excess of free fatty acids be present.

The practical result of these researches is that the *prime essential condition for the prevention of rancidity in fats is complete exclusion of air*, which being secured, exposure to the light or storage in the dark is immaterial.

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CHAPTER III.

PREPARATION OF ANIMAL FATS AND OILS.

THE methods employed for preparing animal fats and oils, that is to say, the treatment of the crude fats, differ somewhat from that pursued for the vegetable fats and oils, but finally conduce to the same end, *viz.*, separation from the accompanying extraneous substances or non-fats. In materials yielding vegetable fats and oils we have to deal with fruits, seeds, etc., forming a dry, solid, and occasionally even hard mass, from which the fats can be obtained economically by the application of considerable pressure, or, when in a pulverised or pulpy condition, by extraction with special solvents. The animal fats, on the other hand, are devoid of the characteristic shell encasing the vegetable materials, the fat lying distributed throughout various parts of the animal body and enclosed in, or completely occupying, cellular tissues; this mode of occurrence of the animal fats influences the method of treatment to be employed.

Whilst the oil- and fat-producing seeds and fruits will bear any length of transport with but little change and arrive from distant parts of the world to be worked up, the crude animal fats must be treated with but little delay and, therefore, usually at the place of their production if they are to be prevented from becoming putrescent, whereby the quality of the product as well as the yield would be seriously depreciated. True, there are certain methods of transport which can be employed, as, for instance, in ships fitted with refrigeration

chambers, as in the case of meat brought from America and Australia, by means of which the raw material is kept intact and unchanged; only, these are much too expensive. There is, moreover, every reason why the material should be treated on the spot.

Most of the animal fats are prepared by a simple process of melting (rendering) in suitable pans, and when separated

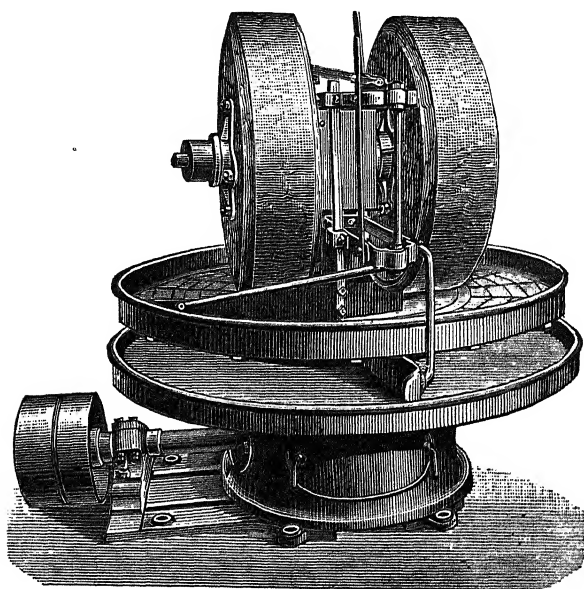


FIG. 1.—Edge runners for crushing fat.

from the residual tissue by straining are ready for use. Other fats, *e.g.*, blubber (fish fat), are partly expressed by the pressure induced when the material is piled up into large heaps, the remainder being recovered—as in some other instances—by boiling in water or by the action of the press. Butter is obtained by a process peculiar to itself, the finely divided fat globules in the milk uniting, under the influence of concussion and agitation, to finally form a lump of fat.

The solvent extraction process which is now employed very largely in the recovery of vegetable fats and oils, on account of the greater yield it ensures, is utilised for animal fats only in individual cases, *e.g.*, the recovery of bone fat and of the residual tallow from greaves.

In consequence of the peculiar composition of the raw material the mechanical appliances used in the preparation of the fats are different from those for vegetable fats and oils. They consist of:—

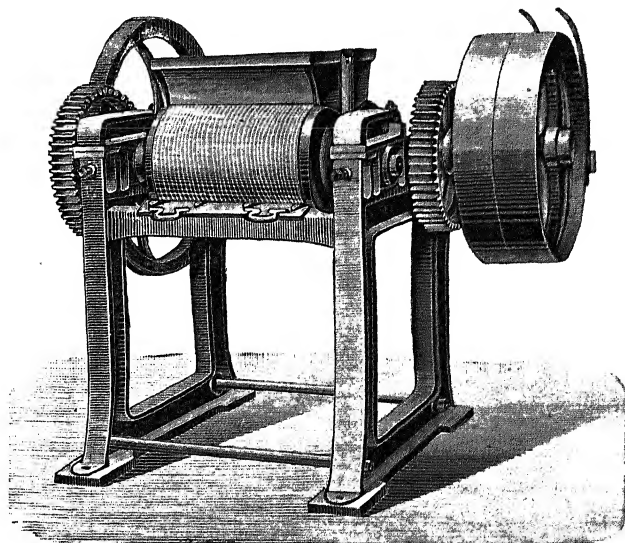


FIG. 2.—Fat-grinding mill (W. Rivoir).

1. Machines for comminuting the fatty tissue.
2. Pans of various construction for melting down the crude fat.
3. Extraction plant for bone fat whereby glue can at the same time be recovered.
4. Presses for pressing blubber, as well as for separating the liquid and solid fats.
5. Filtering apparatus.

Latterly also the operation of separating the fat of fish and marine mammals into their solid and liquid components by centrifugal force has come into practice. The centrifugal machines used for this purpose deliver the liquid fat either over the edge or through lateral apertures in the inner drum, but machines of any known type are suitable.

Those varieties of tallow (raw tallow, core or kidney suet) that are used for making artificial butter are separated into their solid (or oleo) and liquid constituents by heavy pressure, and the so-called press tallow (or beef stearin) is prepared in the same manner by pressing tallow previously refined by melting.

The purification of the animal fats is effected by melting and repeated agitation with water, or bleaching by chemicals—the latter operation being but seldom resorted to. For the removal of small solid particles the fat is liquefied and strained through sieves or filter cloths. Blubber is also treated the same way or clarified by filtration by means of various mechanical appliances, such as filter presses or filtering apparatus of the kinds described in the author's work on *Vegetable Fats and Oils* (*q.v.*).

MACHINERY FOR BREAKING DOWN FATTY TISSUES.

Animal fats are delivered to the melter in lumps of all sizes, sometimes (as in the case of whale blubber) even in large blocks of considerable circumference and thickness; but they cannot be worked up while in this condition, since it would, on the one hand, necessitate the use of excessively large vessels, and on the other, the melting down in an unbroken state would require too much time. However, it is not merely a question of dividing the fat into small portions, but also, and principally, of breaking down and disrupting the cellular tissues in which the fat is enclosed, without which rupture of tissue an insufficient yield of pure fat would be

obtained and the greater part of the material would have to be exposed to an excessively high temperature in order to allow all the fat to exude. The fat is therefore comminuted before melting, an operation which can be effected in various ways. The simplest but most cumbrous and tedious method is by chopping on a block with a sharp knife or hatchet. A better plan is to employ a knife, movable vertically through a fixed point—a kind of chopping machine; but this also is

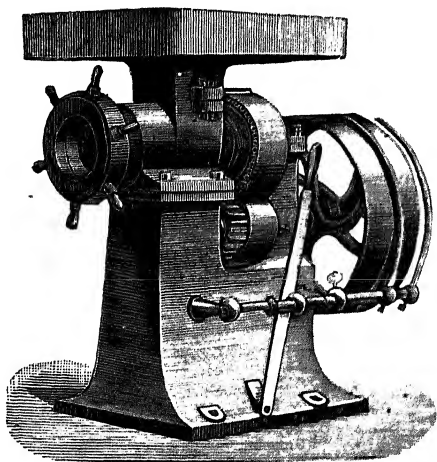


FIG. 3.—Fat-cutting machine with revolving knives.

insufficient for working on a large scale. In England edge runners (Fig. 1), *i.e.*, heavy millstones, made to revolve over a horizontal surface by means of a driving pulley (placed above or below), are used, which crush the fat by their weight, the membrane of the cellular tissue being thereby ruptured and the fat converted into a pulpy mass having a considerable degree of fineness.

Roller mills may also serve for crushing fat, but the fat-grinding machine shown in Fig. 2 is more efficient.

This machine consists of an iron frame on which are

mounted grooved (sharp-edged) rollers driven by toothed gearing and belting pulleys, and at the top a wooden box or hopper for feeding the fat to the rollers. When the rollers are set in motion and the fat placed in the box, the rollers (which move in opposite directions) draw the fat in, cut it and crush it, and then allow it to drop into a receiver situated

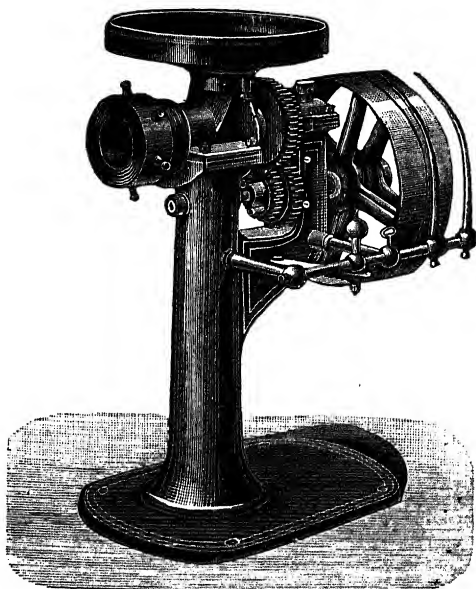


FIG. 4.—Fat-cutting machine with revolving knives.

beneath. In this case the material is not only torn apart but comminuted as well.

The most efficient of all, however, are the cutting machines, such as are shown in Figs. 3 and 4. In the hollow of the cast-iron stand a number of sharp-edged knives are placed, which revolve, and thereby rapidly cut in pieces the material fed from the upper dish, and deliver it by its own gravity into a receiver below.

PANS AND APPARATUS FOR FAT MELTING.

The pans for melting the fat may be selected in accordance with the requirements of the business; these may be of a simple kind if it is merely a question of small quantities or of such kinds of fat, *e.g.*, hog fat, as evolve no malodorous vapours, or, finally, when the works are situated in a place where the neighbourhood suffers no inconvenience by the production of a strong smell, *e.g.*, blubber-melting works in

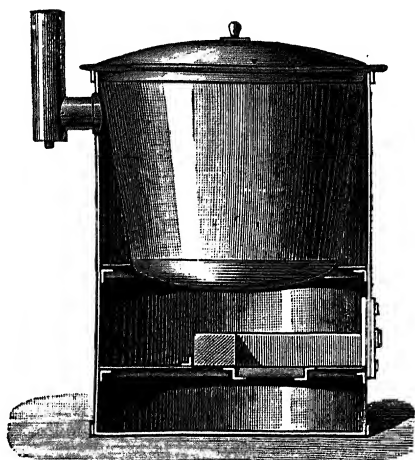


FIG. 5.—Portable melting stove with pan.

the vicinity of the fishery. The pans may be mounted either on portable stoves, as, for instance, in blubber refining, or else surrounded by brickwork, and, therefore, fixtures. In either case, however, the hot gases only and not the flame should come in contact with the bottom of the pan, in order to prevent the melted fat becoming overheated, a condition which would result not only in the discoloration (browning) of the fat, but also cause danger from fire. Iron or wooden vats heated by a steam coil, or jacketed pans, may also be advantageously employed.

A portable melting pan is depicted in Fig. 5, consisting of a sheet-iron stove with furnace door and flue and a wrought-iron or copper pan. The dome-shaped bottom of the pan is protected (around the spring of the dome) from the action of the fire by means of a partition wall.

A fixed pan heated by direct fire is shown in Fig. 6. The pan stands above the level of the bottom of the furnace, and

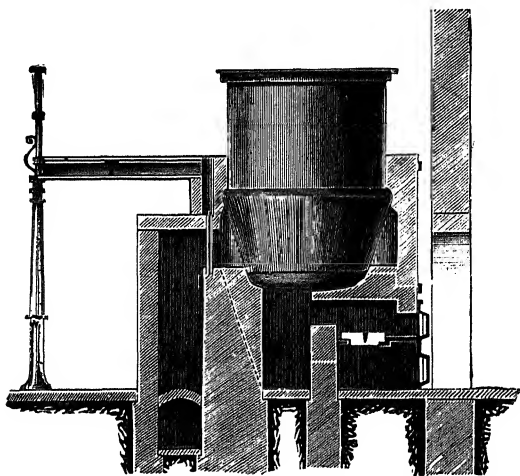


FIG. 6.—Fixed pan heated by direct fire.

is accessible by means of steps. The melted fat can be run off through a tap near the bottom of the pan.

In large establishments, especially in inhabited districts, tallow melting in open pans is prohibited by the authorities, the use of closed vessels being imperative. A number of these appliances are described and illustrated in the following pages.

TALLOW-MELTING PLANT DESIGNED BY TONCOU.

The inodorous tallow-melting plant devised by Toncou is depicted in Figs. 7 to 9. The installation comprises sixteen

pans A, each of which is fitted with a hood B (Fig. 9), together with a tabular superstructure and a manhole C for filling and emptying the pan. The vapour catchers (hood, etc.) B are connected with a draught flue H, about 30 in. high and 26 in. wide, which conveys the vapours under the white-hot dome of a small furnace (situated directly beneath

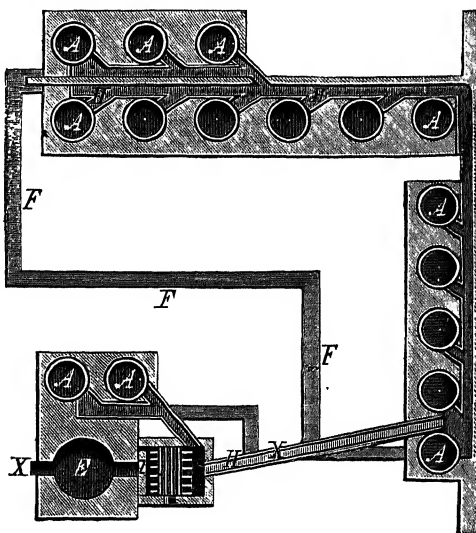


FIG. 7.—Tallow-melting plant heated by direct fire and fitted with deodorising furnace.—A, melting pan; B, hood; C, manhole; D, furnace for each pan; E, vapour catcher; F, flue common to all the furnaces; G, supplementary furnace; H, draught flue leading to G; J, draught hole; K, grating; L, fireproof arch; M, register.

a shaft E, which is about 110 ft. high), where they are completely consumed. The whole of the furnace fires are connected with the aforesaid shaft by a common flue F. In very large works a draught hole J is provided, through which air enters and dilutes the vapours, which then pass through a grating K into the furnace C, the products of the combustion going on in the latter streaming through a grating

into the fireproof arch L, and thence into the shaft. The hearth of the furnace G is 36 in. wide and 47 in. long. Coke fuel is employed here in order to reduce to a minimum the water vapour in the products of combustion. M is a vertical register or damper for shutting off the connection between the furnace G and the shaft E. In the same way the horizontal damper N serves to open up communication,

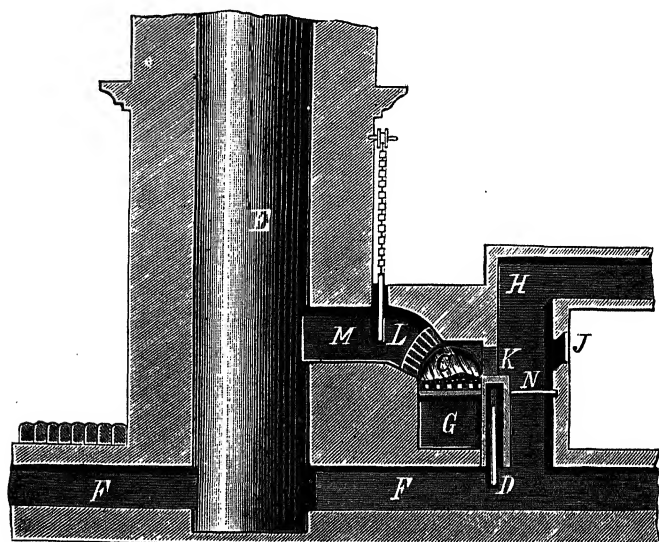


FIG. 8.—Tallow-melting plant with direct fire and deodorising furnace.

when desired, between the flues H and F in order to divert the vapours from the pans whilst the fire is being kindled in G. By means of a second vertical damper O the furnace gases can be diverted through the fire G, the horizontal damper N being assumed as open for that purpose. This arrangement presents the great advantage that the vapours and gases to be burned need not pass through the fuel, the place where the coal is consumed being distinct from the chamber wherein the actual destruction—combustion—of the

gases is effected, so that, since pure air alone is admitted to the fuel, the fire can be made stronger or reduced at will. It goes without saying that the gratings K and L must be made of fireproof material (firebrick). Notwithstanding this, the brickwork easily becomes damaged on account of the large quantity of moisture contained in the vapours.

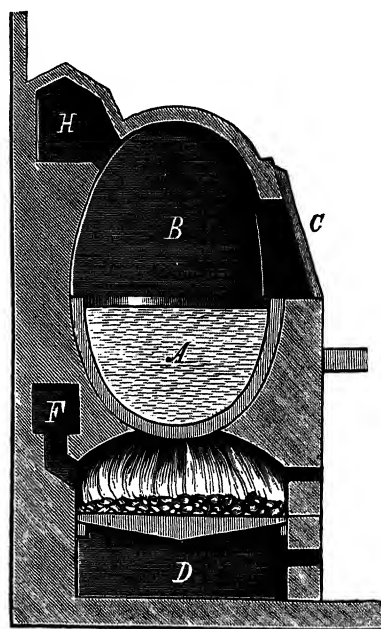


FIG. 9.—Vertical section of a melting pan.

TALLOW-MELTING PLANT FOR SULPHURIC ACID METHOD.

In order to obviate the numerous inconveniences attendant on the melting of tallow in open pans, and the great expense of large installations for working without smell, a practice has latterly arisen of melting tallow with the assistance of dilute sulphuric acid. An apparatus designed by Vohl for this purpose is shown in Fig. 10.

This consists of a lead-lined, cast-iron pan A, with cylindrical cover B and lid C, fitted with a mica plate D. A similar plate is fitted in the door E, and by this means inspection of the course of the operation proceeding inside the pan is facilitated. At night an artificial light is placed over the mica plate in the lid C. Mica is selected for these windows on account of its greater durability and security as compared with glass. The door E serves for the admission of the

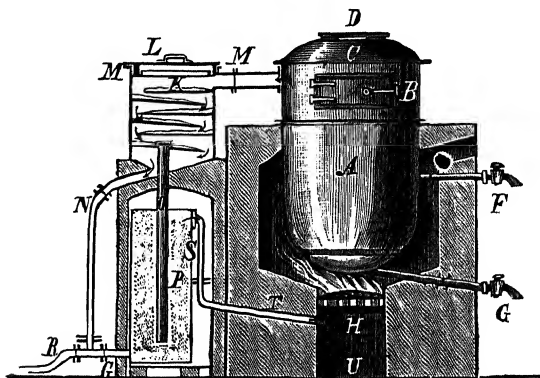


FIG. 10.—Tallow-melting plant with direct fire (for acid treatment).—A, lead-lined, cast-iron pan; B, cylindrical lid; C, domed lid; D, mica plate; E, door; F, outlet tap for tallow; G, tap for drawing off the acid liquor; H, hearth; J, pipe leading from pan to condenser; K, condenser box; L, lid; M, packing round the rim; N, outflow pipe; O, effluent for the easily condensable gases; P, condenser; Q, outflow pipe; R, outflow; S, outflow pipe for the permanent gases; T, flue; U, ash-pit.

tallow and hermetically closes B, besides serving for withdrawing the greaves left on the grating at the bottom of the pan when the operation is terminated, after the tallow has been run off through the tap F and the acid liquor through G. The gases and vapours generated in A during the melting process pass through the pipe I to the condenser K, which is closed by a lid L packed round the edge M. Inside, the box K contains a number of inclined platforms covered with slaked lime. The box itself is made of wood impregnated

with tar and asphalt. The condensed water runs away through the pipe N and forms in the swan-neck R a water-seal preventing the escape of vapour. The uncondensed gases and vapours issue from K *via* the pipe O into the lead-lined condenser P, which is filled with coke or pumice, impregnated with sulphuric acid, and the accumulated liquid runs off at the bottom through the pipe Q to the discharge pipe R, the permanent gases, etc., being finally led through the pipe S into the conduit T which debouches in the ash-pit U below the hearth of the furnace H, where they are consumed. These gases can be delivered underneath the fire without any fear of reducing the draught, since they have been mostly freed from water in the condenser. The ash-pit U is fitted with an iron door by means of which a powerful draught can be induced for exhausting all the gases from the apparatus and carrying them to be consumed in the furnace. V is a funnel leading to the shaft and effecting the removal of the furnace gases.

In working this apparatus 20 parts of water previously mixed with $\frac{1}{2}$ to $1\frac{1}{2}$ parts of concentrated sulphuric acid are employed for each 100 parts of tallow to be melted. The sulphuric acid serves to effect the chemical decomposition and bursting of the cells.

WILSON'S TALLOW-MELTING APPARATUS.

In the Wilson apparatus (Fig. 11) the steam enters through the perforated pipe G beneath the false bottom. The plate F fits tightly over the aperture E and the vessel is charged with tallow to two-thirds of its capacity. The steam pressure may be as high as $3\frac{1}{2}$ atmospheres, a pressure which is maintained during ten hours. The condensed water collects beneath the false bottom and can be drawn off when required. The melted tallow runs away through the taps P, P, and the greaves are finally removed through the aperture E.

GELLHORN, FLOTTMANN & CO.'S STEAM APPARATUS FOR
TALLOW MELTING.

The fat to be melted is introduced into the melting pan A, through the neck, and rests on a grid B covered with fine filter cloth. The pan is situated in the basement, and the neck projects through the ground floor, an arrangement economising both time and labour in charging the pan. The

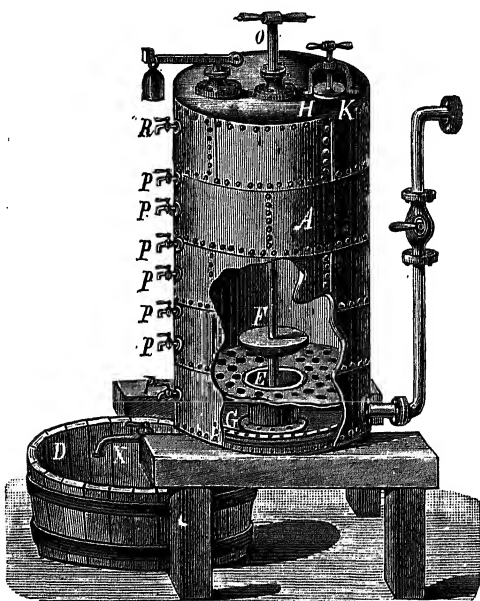


FIG. 11.—Wilson's tallow-melting apparatus.

latter is constructed of strong sheet-iron, jacketed with a tin case and slag wool lagging to prevent loss of heat by radiation. When the melting pan A is filled with crude fat, water is poured in until the charge is covered, and the neck is then closed by a steam-tight lid.

Steam is conducted from the boilers C through the pipe D into the melting house as far as the pan A, where it passes

less than that in the pan. To prevent the steam escaping from the latter back into the boiler by way of the feed-pipes E, D, a back-pressure valve J is affixed in D. A and D are also provided with pressure gauges to enable the pressure to be observed. When the differences of pressure are considerable the valve G is closed. Also at the end of one hour the

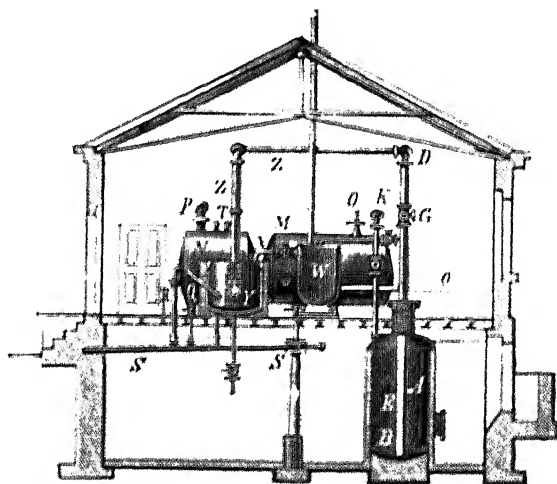


FIG. 14. Gellhorn, Plattmann & Co.'s plant (section through melting house).—A, melting pan; B, grid with filter; C, boilers; D, steam pipe; E, F, branch therefrom to A; G, H, valves in pipes E, F; J, back-pressure valve; K, pipe to condenser; L, valve in K; M, condenser; N, receiver; O, water-pipe; P, pipe conveying the gases to the furnace; Q, R, discharge pipes; S, general outflow pipe; T, manhole; U, pipe from A to W; V, valve in pipe U; W, clarifying vat; X, conduit from W to Y; clarifying pan; Z, steam pipe leading to Y.

valve G is shut and the valve L in the pipe K opened, whereupon steam and the malodorous gases evolved by the crude fat pass, in company with contained globules of fat, from A into the condenser M, which, together with the attached receiver N, is half filled with cold water. The pipe K is continued within the condenser M and debouches below the level of the water therein. Sufficient water is supplied by the pipe

O to condense the vapour and fix the malodorous gases in the water, whilst the uncondensed gases pass with the fat globules and warmed water into the receiver N, and the gases that do not remain in the latter then pass through the pipe P to the back of the fire bridge in the boiler fire, where they are consumed. The inclined tube V in the receiver N serves to keep the water-level in M and N constant by drawing off a quantity of warm water equal to that of the cold supplied and delivering it into the pipe R, which discharges it through the common outfall S into the open air, where it can be mixed with soil for manure.

The fat accumulating in time in the receiver N is removed through the manhole T. Taps leading to the outfall pipe S are fitted to the receiver and condenser in order to draw off their contents when they have to be emptied and cleaned.

Condenser, receiver and melting pan are built of strong sheet-iron. When the gas is all removed the supply of water to the condenser is arrested, and the pan A disconnected from the condenser by closing the valve L. G being reopened, fresh steam is admitted into A, and the crude fat is again melted, this time for four hours, whereafter G is once more closed and the vapour and gas again passed through the condenser as already described. When the gauge on the pan shows that all the pressure is removed, A is closed and left for an hour, during which period of repose the preliminary clarification of the melted fat from the admixed solid residue and dirt is effected. The valve H in the pipe F being then opened, steam enters the upper part of the pan and presses on the surface of the fat there floating on the water; and when the valve V is opened the fat and water are forced through the pipe U into the clarifying vat W, the water entering first and being followed by the tallow, which is freed from the solid residue and coarser particles of dirt by the filter B, these latter being removed from A through the lower opening, which at other

times is tightly closed. The clarifying vat W is of sheet-iron and has the largest possible surface; here are separated the particles of dirt, part of them sinking to the bottom and part forming a thin scum on the surface of the fat. After remaining at rest in this vat for six to eight hours the fat is then gradually floated upwards by means of water, admitted through the pipe O, until it runs off through the tapped pipe X into the clarifying pan Y, the water and deposited dirt in W being drawn off from the bottom. The vessel W is covered in, the vapour which enters when the mass is forced over from the pan A escaping through a pipe extending into the open air above the roof. The clarifying pan Y is a wrought iron jacketed pan, open at the top, constructed to stand the working pressure (3 atmos.) of the steam in A, and lagged with lead and slag wool to prevent radiation. Before the fat is admitted the pan is partly filled with clean cold water, and then, when the fat is in, the mass is gently boiled by means of steam admitted into the jacket space from the pipe D and Z. After treating in this way for an hour, during which time the scum is taken off, the valve in the pipe Z is closed, and the steam and water of condensation are run off through S.

When the mass is so far cooled that it begins to set, the tap in the pipe leading from the bottom of the clarifying pan is opened and the contents drawn off, the water and dirt first, and then the purified fat, which is delivered into the transport casks. In the hot season cold water from O is passed into the jacket space around the pan and assists the setting of the fat.

By means of this apparatus perfectly pure tallow is produced.

LOCKWOOD AND EVERITT'S STEAM PLANT FOR TALLOW MELTING.

The advantages of this plant consist chiefly in the complete destruction of the evil-smelling vapours injurious to health,

and in the security of the apparatus against explosion, the fat being gradually melted in a digester. The apparatus (Fig. 14) consists of two parts, *viz.*, a melting pan and a furnace for consuming the evolved gases and vapours, these two parts being connected by a pipe J. The pan, or digester, which

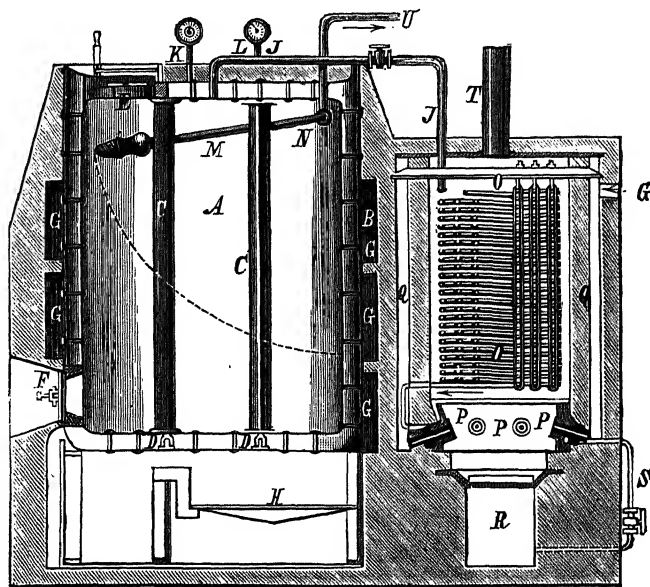


FIG. 14.—Steam plant for tallow melting.—A, sheet-iron pan ; B, jacket ; C, steam pipe ; D, stay rods ; E, manhole ; F, outlet ; G, flue ; H, hearth ; J, pipe leading to furnace ; K, pressure gauge of steam jacket ; L, pressure gauge of digester ; M, exhaust pipe with strainer ; N, swivel joint ; O, system of tubes ; P, burner ; Q, opening and air chamber ; R, hearth ; S, tube ; T, flue ; U, outlet pipe.

contains the fat to be melted, consists of a steam-tight cylindrical vessel A surrounded by a jacket B. In order to make the apparatus extremely firm—since it has to bear a temporary pressure of 7 atmospheres—the top and bottom of the cylinder are connected by iron stays, and the jacket with the inner cylinder by stay bolts, in addition to which the

steam pipe C, which serves to regulate the temperature of the charge of fat, also assists in strengthening the pan. Under the vessel, which is mounted on feet, is a furnace, the hot gases from which pass through flues G cut in the brickwork in such a manner that the gases come in contact with as large a surface as possible of the pan and finally escape into the chimney. The melting pan is filled by way of a manhole H, and the residual skin, greaves, etc., are removed through an opening F. The removal of the liquid fat is effected by an exhaust pipe M turning on a swivel joint N, and fitted at the end with a strainer to keep back extraneous matters, the other end leading to an outlet pipe U, through which the fat, forced upwards by the pressure in the pan, is delivered to any convenient spot. The gases and vapours generated during the melting of the crude fat are led through the junction pipe J to the Argand furnace, as the inventor designates this portion of the apparatus. Here the gases at first traverse a system of heated tubes O and then issue through four burners P arranged symmetrically around the furnace, and are there mixed with atmospheric air and burned. The products of combustion in ascending surround the spiral O and escape into the chimney T. The air requisite for combustion enters at the upper part of the furnace, at G, into an air chamber enclosed in the brickwork, where it is heated, and thence passes to the burners below. In order to increase the draught a small tube S conducts heated gas under pressure into the space below the hearth R. With this description the working of the apparatus can be explained in a very few words. After the jacket B has been filled with water up to a level with the top of the flue, the charge of fat is placed in the digester, care being taken that the exhaust pipe M is first raised to its highest position and fixed there. Simultaneously with the closing of the feed-opening the Argand furnace is kindled, and as soon as a moderate amount of pressure is indicated

by the gauge K the tap in the pipe J is opened and the steam is allowed to escape to the combustion furnace, which in the meantime have become hot enough to ensure the destruction of the malodorous gases. The heating of the melting pan is so regulated that the pressure inside the pan does not exceed $2\frac{1}{2}$ atmospheres and that in the jacket does not more than 4 atmospheres. In order to ascertain when the melting is complete, samples may be taken from the pan at any time through the pipe M. The capacity of this digester is generally $7\frac{1}{2}$ tons. When the residual tissues are intended for fodder they are dried at night in the pan itself, a low fire being kept up in the combustion furnace.

STEAM APPARATUS FOR TALLOW MELTING.

A simple form of tallow melter heated by steam is shown in Fig. 15.

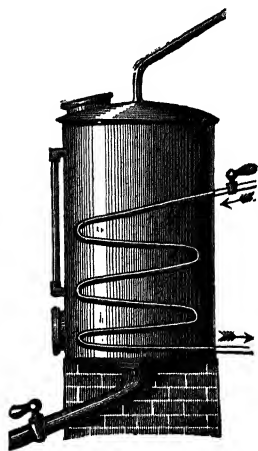


FIG 15.—Steamer for melting tallow.

The apparatus consists of a vessel covered with a hood which is provided with an effluent pipe. The steam through which steam at a pressure of several atmospheres is admitted is wound in spiral form in such a manner that the upper tier of the coil is situated at a level about one-third the height of the pan, the steam being directed progressively downwards until the lowest one makes exit at the foot of the vessel. The steam enters at the top of the coil and condenses to water therein necessary to give the coil such a

that the water can run away freely. At the commencement of working the steam is regulated so that water alone flows from the lower extremity of the coil, and the steam current

allowed to act until the crackling sound caused by the evaporation of water from the greaves is heard inside the pan.

To facilitate the removal of the melted fat the bottom of the pan is made somewhat concave and is fitted with a pipe, above which is a grid supporting the greaves, these latter being afterwards removed through a lateral opening (covered by a screwed lid) on a level with the false bottom. A glass tube gauge, fitted at the side of the pan, enables the height of the melted tallow in the vessel to be observed. The pan is also lagged with wood to prevent the radiation of heat.

A slow current of steam may be passed through the coil even while the pan is being charged, the current being turned on more fully, as already described, when the pan is closed. Directly the fat is melted the steam is shut off, the fat being then run away through the pipe at the bottom of the vessel, which latter is refilled with tallow after the greaves have been removed.

RIVOIR'S STEAM APPARATUS FOR TALLOW MELTING.

This apparatus, displayed in Fig. 16, works without emitting any smell, the liquefaction of the fat being effected by steam under a pressure of from 3 to 5 atmospheres. The pan is made of wrought-iron, with a riveted, rounded bottom, and stands upright on a wrought-iron frame. In the lower part of the cylinder two perforated false bottoms are fixed at a little distance apart, the fat to be melted being laid on the upper one, which is readily accessible from without through a lateral aperture closed by means of a bow and screw and situated on the same level with the false bottom. This opening serves for clearing out the greaves. Between the false bottoms there is a lateral cock for drawing off the melted fat.

Beneath the lower false bottom is situated the steam coil, and below that again, in the deepest part of the pan, are the pipe and cock for emptying the vessel completely. At the

top of the apparatus is a manhole, fastened by a bow and screw, through which the pan is filled, and entered for cleaning ; and an escape pipe for the vapours is also situated in the top of the vessel. When the apparatus is connected with a steam supply working at a pressure exceeding 5 atmospheres it must be provided with a safety valve, pressure gauge, reducing valve and back-pressure valve between the

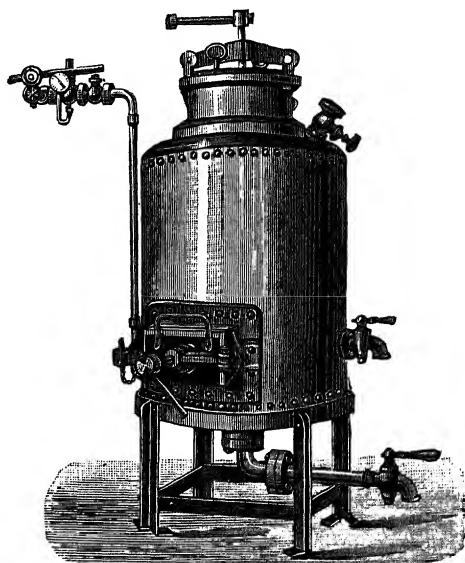


FIG. 16.—Steam apparatus for tallow melting (W. Rivoir).

pan and the feed-pipe. Where steam is not available the apparatus can be modified so that it can be set in brickwork and generate its own steam for the melting process.

For working with indirect steam, *i.e.*, without the steam and fat coming into direct contact, the maker supplies jacketed pans well tinned inside, the heat being then applied by a steam coil situated in the space between the two walls of the pan. A cold water coil is also provided, so that the temperature can be regulated as desired. The interior fittings

comprise a vertical pipe and a draw-off cock as in the case of the single pan, a cock for completely emptying the vessel being placed, with its attached pipe, centrally, at the lowest point in the bottom. The pan itself is supported on a wrought-iron stand.

In order to accelerate the melting process stirrers are provided which squeeze the lumps of melting fat between arms. These stirrers are made of well-tinned wrought-iron and are easily taken out for cleaning. Motion is imparted from a driving pulley above and a pair of cone wheels, and they can be arranged for driving either by hand or power.

By reason of their simplicity and great capacity Rivoir's melting pans have proved exceedingly useful, and are preferred by experienced melters before more complex and less practical systems.

For melting good quality tallow, such as *premier jus*, or edible fat, Rivoir supplies simple, round, wooden vats heated by direct steam, which is admitted through a coil at the bottom. A draw-off cock for the melted fat is placed at a moderate height above the base, the fat being delivered to this cock by a jointed pipe, an arrangement which allows the clear fat to be drawn from any level within the vat. A cleaning-out valve is fitted in the bottom, the aperture being made sufficiently large to enable the residue to be removed from the vat with ease and the latter swilled out with water. All the ironwork coming in contact with the fat is well tinned.

O. HEINTSCHEL'S TALLOW-MELTING, CLARIFYING AND FILTERING APPARATUS FOR CONTINUOUS WORK.

The apparatus shown in various points of detail in Figs. 17 to 20 consists of three parts: the melting pan A, the clarifier B, and the filtering vessels C_1 C_2 C_3 .

The melting pan is a cylindrical vessel with a conical bottom, which, as shown in Figs. 17 and 18, is jacketed, the

intermediate space between the walls containing apparatus H_1 , composed of coiled or transverse pouches. In the axis of this pan is situated a vertical cylinder F_1 (Fig. 19), projecting through both lid and bottom of the pan and fitted with stirrers and overhead drive. Being open below and projecting through the bottom

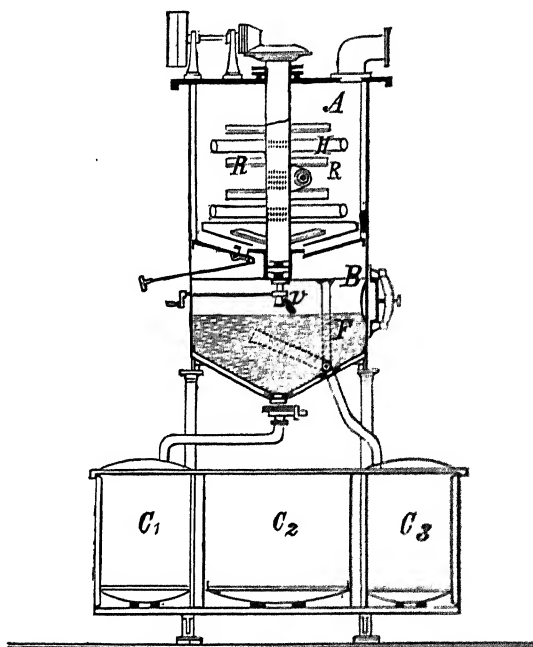


FIG. 17.—Heintschel's tallow-melting, clarifying and filtering apparatus. (Front vertical section.)

cylinder communicates with the second chamber closed by means of a slide or trap, etc., V , whereby chambers can be isolated. Close to the filter tube at the deepest point of the bottom of A , is a second opening (Fig. 19), so that a valve adjustable from outside enables the apparatus to be connected with or shut off from one another. Each filtering cylinder has a perforated jacket, whereby t

troduced into A through the feed-opening E is, after being melted by the warm air in the double walls or heating apparatus, enabled to filter through the openings in the said cylinder. When V (Fig. 18) is open the fat drains away and collects in B. In order to increase the filtering capacity the tube F may be replaced by perforated filter tubes, the perforations being made only on the side of the tube opposed to the direction of movement of F, in order to prevent the clogging of the filtering surface. For the same purpose a slide, perforated with apertures corresponding to those on the cylinder, can be mounted thereon, and by being moved up and down be made to clear away any fibres, etc., attaching themselves to the apertures; or a scraper may be affixed to part of the apparatus so that its cutting edge rests on the filter cylinder and scrapes off any adherent matter during the rotation of the cylinder.

The clarifying chamber B is situated underneath or by the side of A, and, like the latter, has a deepened bottom and a heating apparatus. At the deepest point in the bottom it is fitted with a closable aperture, and near this a telescopic or hinged tube (adjustable from the outside), or else a number of taps at different heights. When a sufficient quantity of melted fat has been run off from A into B then the chambers are isolated by closing the aperture of communication. In consequence of their different specific gravity, the various grades of tallow, as well as the admixed particles of fibre and water, quickly separate in layers, the better, lighter grades being at the top, the others successively lower, and the water and

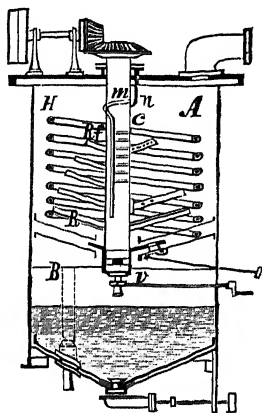


FIG. 18.—Heintschel's apparatus. (Detail's of melting pan.)

fibrous particles weighted therewith lowest of all in the deepened bottom B; so that by suitably adjusting the draw-off tube, or by way of the taps, definite grades of melted fat can always be run off from above downwards and conducted by the flexible tube D into the clarifying vessels.

The solid constituents (greaves) collecting, in the course of

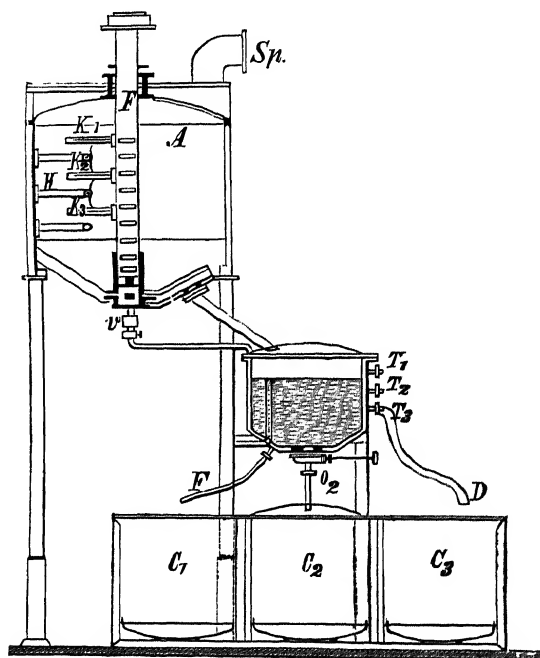


FIG. 19.—Heintschel's apparatus. (Lateral section.)

the continuous melting process, in the bottom of the melting chamber A, as well as water, blood residue, etc., are drawn off from time to time through O into the empty clarifying chamber, where they are clarified, the hot liquid fats they still contain being removed through T; and, finally, the less valuable, thick residues are withdrawn through O into the vessels placed underneath, where they can be subjected to further treatment.

In this process, by means of the continuous series of operations performed in succession (melting, clarifying and drawing off), the capacity of the apparatus is far in advance of the old-fashioned plant as regards both the quantitative and qualitative results obtained. Moreover, this system possesses the additional advantage that by drawing off through the adjustable pipe B and collecting extremely uniform grades of melted fat in the clarifying vessels, a well-assorted class of goods is produced and the raw material utilised to the best advantage. In order to enable the separation of the fats to be effected according to specific gravity in the chamber A_1 , another construction is given to the filtering cylinder. This is perforated with a convenient number of holes or slits arranged in rings at different heights and is provided with a well-fitting inner tube perforated to correspond.

Thus, for example, if the fat is to be separated into three portions at equal intervals of height, three equidistant rows of rings are provided. By raising or lowering the tube, *e.g.*, through a distance equal to one-third of the interval between the rings, the sifting rings opposite the one layer are brought into juxtaposition, the openings in the other two being covered and thereby closed. By this means a qualitative separation of the melted fats can be effected even in the melting chamber A, uniformity of quality being thereby more effectually ensured.

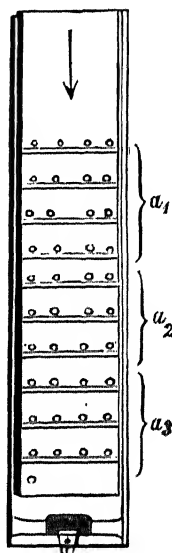


FIG. 20.—Heintschel's apparatus. (Filtering cylinder.)

FAT-EXTRACTING APPARATUS WITH CORRUGATED BOTTOM.

The apparatus shown in plan in Fig. 21, and in side and front view in Figs. 22 and 23 respectively, is designed for

melting fat or extracting it from fatty substances. It is enclosed on all sides and heated internally by steam passing through several series of parallel pipes *a* arranged at different

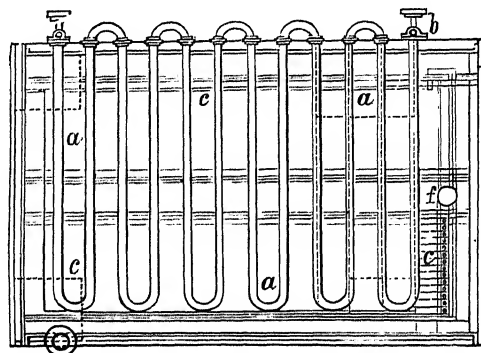


FIG. 21.—Fat-extracting apparatus with corrugated bottom.

heights, the walls being covered with insulating material to prevent radiation. The arrangement is such that the steam from a common feed-pipe *c* enters each series at the same

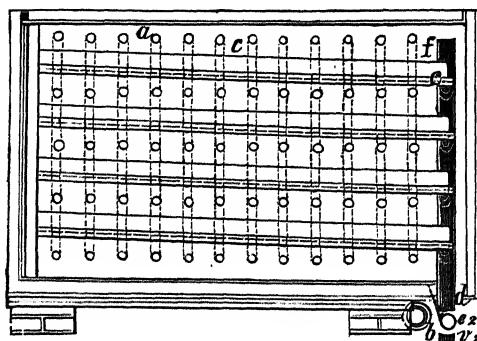


FIG. 22.—Apparatus with corrugated bottom. (Side view.)

time, so that a uniform temperature is maintained throughout all parts of the tubes and apparatus. In order to increase the influence of the heat, radiated from the pipes, on the fat or fatty matter in the flat boxes *c*, and, with this object, to

obtain the largest possible extent of surface, the bottom of the box is made of corrugated tinplate, an arrangement greatly increasing the area of surface exposed in comparison with a flat bottom. In the hollows formed by the corrugations are fixed semicircular or angular strips of iron, which are perforated all along the edges, so that the melted fat can run through into the channel formed by the iron strips and hollows of the bottom and run down to the front of the box, which is laid at an angle for this purpose. Not only is the draining of the fat facilitated by this arrangement but the

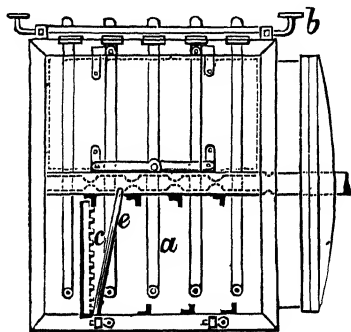


FIG. 23.—Melting apparatus with corrugated bottom. (Front view.)

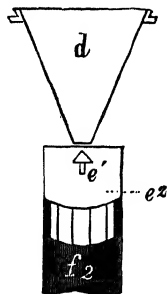


FIG. 24.—Arrangement of Funnel.

impurities are also retained. The fat arriving at the front of the boxes runs down through the apertures there into open channels *e*, which convey it to a vertical collecting pipe. In this case the corrugations extend to the front end of each box. Instead, however, of having a channel *e* underneath, the front edge of the box may be inclined towards the centre of the apparatus, and when this is the case the corrugations do not extend quite to the edge, but a ledge sloping down towards the middle of the apparatus is attached thereto. The fat coming from the channels or boxes runs into this ledge and is conveyed therein to the centre of the apparatus.

Under these ends are arranged stays (fastened near the collecting pipe) which catch the fat and lead it into the collecting pipes, from whence it passes to the collecting vessel. Another method of draining the fat from the front end of the boxes consists in allowing it to fall direct into a channel at the bottom of the apparatus, in which case the ends are arranged stepwise, the upper ones always projecting beyond those successively lower, so that the fat can drop direct from each to the channel e^2 , Fig. 24, which slopes down towards the middle and terminates there in a collecting pipe. To prevent the fat splashing as it falls into the channel the latter is made in the shape of a funnel, with an angle as acute as possible, so that splashing is out of the question. This channel may either be provided with a small lateral opening discharging into a concave channel e^2 , or may have an opening at the bottom, under which a partition wall e^1 , covered with a shed roof, is arranged.

Instead of fitting the boxes with corrugated bottoms as described, ordinary melting vessels may be employed, corrugated plates being fitted either on the bottom or at a slight distance above. The fat is placed on these plates so that the melted fat flows down the channels and collects in the bottom of the vessel, to be then drained away in a suitable manner.

SOLVENT EXTRACTION PLANT.

As already mentioned, the solvent extraction process, which in the vegetable fat and oil industry is of considerable importance, is employed only in isolated cases for animal fats, the nature of the latter being different. Bone fat is the fat mostly recovered by extraction, and rationally conducted establishments either extract the fat alone from the bones and treat the residual matter in other ways, or work up the bones completely in one operation. The forms of apparatus

described in that section of the author's work on *Vegetable Fats and Oils* dealing with extraction processes (*q.v.*) are also applicable for the treatment of bones as well as of the residue (greaves) from fat-boiling works, whilst the plant now described is intended specially for the extraction of bone fat.

DR. AHRENS' APPARATUS FOR EXTRACTING BONE FAT.

Latterly it has been proposed to replace the benzine, almost universally employed for depriving bones of their fat, by carbon tetrachloride, which is a powerful solvent of fat, has a constant boiling temperature, is unflammable and leaves no unpleasant smell in the fat after saponification. If, however, carbon tetrachloride be used in the ordinary fat-extraction apparatus a remarkable phenomenon is observed: the fat being either black or very dark brown in colour. This results from decomposition products of carbon bisulphide, which is always present in commercial tetrachloride, the bisulphide forming, with the ammonia in the bones and iron in the blood, sulphocarbonates, which decompose into carbonates and sulphur compounds, which remain behind in the extracted fat after the removal of the solvent by evaporation and thus impart to it a dark colour. On the other hand, chemically pure carbon tetrachloride cannot be employed, on account of its high price.

Nevertheless, the present process and the apparatus therefor enable the ordinary tetrachloride, contaminated by carbon bisulphide, to be used, the above-mentioned objection having been overcome. The process consists in depriving the bones of their fat in an open extractor fitted with two perforated false bottoms and a cooler, the cooling water of which covers the bones and the solvent. The ammoniacal vapours expelled from the bones during the heating of the solvent, form, with the accompanying air, bubbles, which ascend through the liquid and carry with them the carbon tetrachloride. In

tetrachloride is run in from the storage vessel R until the bones are covered and the solvent attains the level of m , which can be ascertained by means of the windows. The tap a_2 of the pipe supplying the solvent is then closed and water is poured into the cooler B through a neck s until the level of the upper manhole is attained. Meanwhile the valve a_1 of the overflow pipe u is closed, the manhole cover being off. The solvent in A is then heated to boiling-point by a steam coil B_1 under the lower grating (false bottom) m and dissolves out the fat contained in the bones. The vapours of carbon tetrachloride evolved during the boiling ascend into the water in the cooler, are there condensed and fall back into A, to then renew their course. At the commencement of boiling, air and ammonia are given off from the bones and carry some tetrachloride with them. The partition walls Y in the cooler B, which serve to separate the

bubbles of gas, run in a spiral direction upwards, inclining towards the axis of the cooler and being connected together by fine sieves. The ascending bubbles impinge on the spiral walls and are diverted laterally, passing from one compartment into another until they burst. By the impact of the bubbles on the partition walls and their passage through the sieves, the tetrachloride is, under the concomitant action of the cooling water, deposited, and, with the assistance of the stream of water trickling from S, rolls down the steep plane until it reaches the lowest point, whence it is diverted back into the extractor. The ascending ammoniacal vapour is dissolved by the cooling water and is conveyed away through the overflow pipe u for further treatment. This overflow pipe is fitted with a

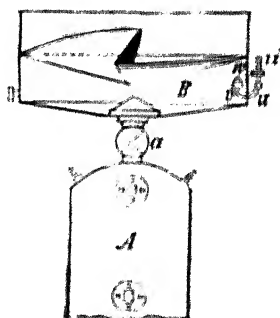


FIG. 26.—Ahrens' bone fat extracting apparatus (Cooler.)

cover in the vicinity of its embouchure in order that the descending tetrachloride may be diverted sideways. Any tetrachloride carried away, notwithstanding this arrangement, is removed by a separating funnel D fixed at the lower extremity of the overflow pipe, where it falls to the bottom and is conveyed through a short pipe *d* into the conduit *c* and into a circulation vessel, whilst the cooling water runs away from the separating funnel.

When, after several hour's exposure to the tetrachloride, the bulk of the fat is dissolved, the solution is run off through the tap *a* into the still *c*, and the extractor re-charged (after closing *a* and opening *a*₂) with solvent from the vessel B to the same height as before. The solvent is distilled off in C and is condensed in the cooler K, whence it flows through *e* and E back into the extractor. The circulation vessel E, which is connected to the cooler K on the one hand and with the extractor on the other, serves to control the progress of the operation, in that the liquid distilled from the fat flows into it (E) from the cooler and thence into the extractor. For the purposes of this control a water-gauge glass is attached to the circulation vessel, so that by observing the fluctuations of the level of the liquid in the gauge the course of the operation can be judged.

The extraction of the fat is gradually effected in the foregoing manner, and the solvent becomes progressively poorer in fat. As soon as a sample taken from *c*, between the extractor and the still, is found to be free from fat the extraction is finished, and the expulsion of the solvent from A is proceeded with. This is accomplished by admitting direct steam into A through pipes *a*⁴ by closing the valves *a* and *c*, turning the steam valve *k*¹ back to *k*, drawing off the cooling water through the pipe *b*², closing the manhole lid *b*, and then opening the tap *b*³ of the pipe leading to the cooler. Hereupon the solvent passes over into the cooler K, where it is

condensed and returned to the vessel R for use over again, whilst the fat is freed from the last traces of solvent in the still C and is finally drawn off. Several of these extractors can also be united to form a battery, and the solvent remaining in the first extractor at the conclusion of the operation may be transferred to the second extractor ready filled with bones, an arrangement saving both time and steam. Moreover, at the commencement of operations the (closed) extractor can be connected with an air pump in order to exhaust the bones of air and gas and thus reduce the amount of gas evolved; this without deviating from the principle of the invention.

In Fig. 26 the cooler B is modified in so far that it is flattened at the base and expanded laterally and connected with the extractor A by a shut-off valve *a*⁹. The cooler may be also jacketed or fitted with a worm tube to reduce the temperature.

By the method of extraction in open vessels with carbon tetrachloride—boiling at 65°-78° C. according to its degree of purity—the employment of a uniform low temperature, and consequently the production of good fat, is rendered feasible, the decomposition of the solvent being, furthermore, prevented, since all the evolved gases and salts are removed by the supernatant cooling water. In addition to this the glue in the bones is greatly protected by reason of the low temperature employed and of the insolubility of the glue in tetrachloride.

J. KALECZOK'S BONE-FAT EXTRACTING APPARATUS.

The vessel *a*, which is fitted with a perforated false bottom, is filled with bones, and the opening *b* closed. Hereupon, the hemispherical steam pan is placed under steam pressure, and the tap *d* in the feed-pipe supplying the solvent (benzine) opened. The benzine flows from the feed-pipe *b* on

to the hemispherical surface, where it is vaporised, and ascends, along with the vapours given off by the bones, through the pipe *e* into the condenser *f*. The condensed solvent and the water (the steam from the bones) arrive at the chamber *h*, whence the water runs off through the pipe *i*, and the benzine continues on its way through the chamber *g* and thence

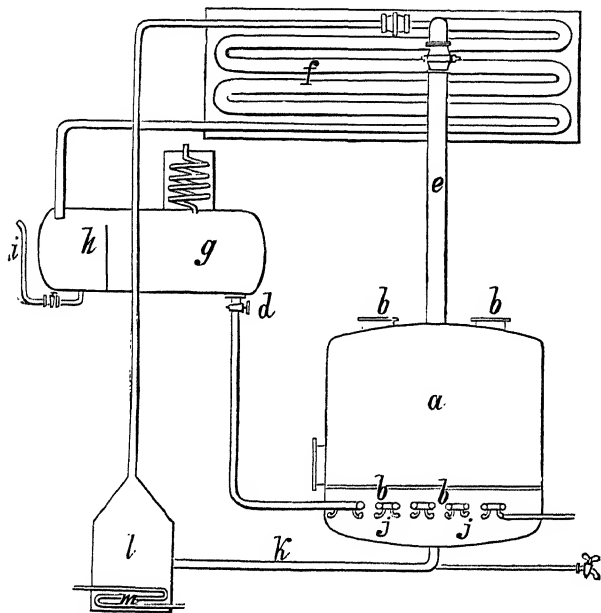


FIG. 27.—Kaleczok's bone-fat extracting apparatus.

through the feed-pipe back to the vaporising pan. The fat extracted from the bones, accompanied by a small quantity of the solvent, runs away from *a* through the pipe *k* to the vessel *l* containing the steam coil *m*. The steam pipe in the bottom of *a* is arranged as a spiral coil.

A screen fitted over the benzine supply pipe prevents the extracted fat from coming into contact with the hot steam pipe. A number of small apertures in the benzine pipe per-

mit the outflow of the solvent on to the hemispherical surface of the vaporising pan. The effluent liquid is immediately vaporised, so that overflow is impossible. A flange, which serves to support the feed-pipe, forms the connection between several branch tubes.

THE HOLDHAUS APPARATUS FOR EXTRACTING FAT AND DRYING THE RESIDUE.

The apparatus displayed in Fig. 28 serves for the extraction of fat from tissue and also for drying the residue. It

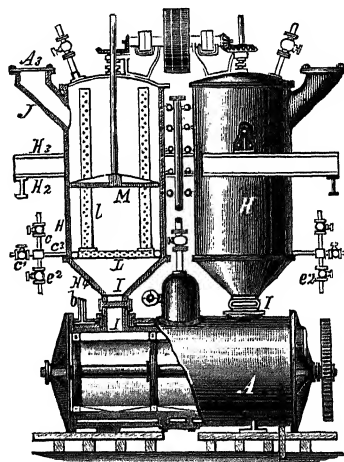


FIG. 28.—The Holdhaus fat-extracting apparatus.

consists of a drying cylinder A, provided with an arrangement for warming and manipulating the contents. A series of cylindrical vessels H is superimposed, and these communicate with the drying cylinder by apertures I in the bottom. These vessels are also provided with suitable openings for the admission of the material into the recipients, and each is fitted with a workable piston M, which can be forced downwards by the mechanism provided for that purpose, thus squeezing the liquid out of the boiled material. The liquid is

then drawn off through the valve e^2 . The vapours from the vessels H are condensed and removed by means of one or several condensers and suitable pipes. The pressed material is then transferred from the vessels into the drying cylinder, where it is freed from moisture.

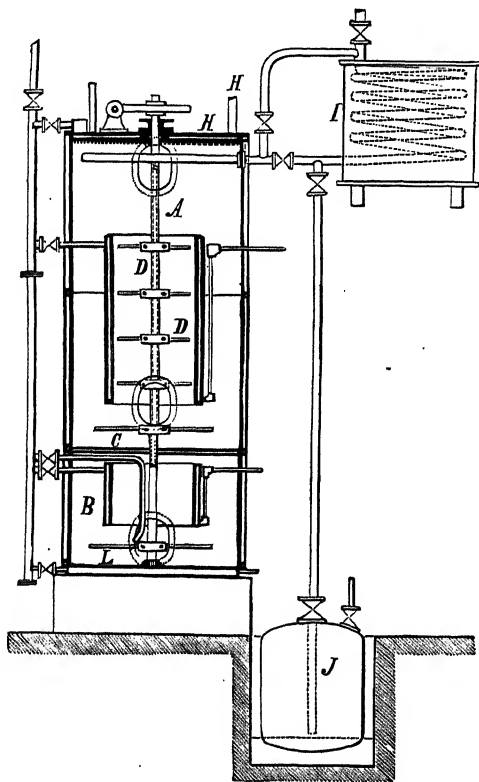


FIG. 29.—Meikle's extractor for bones, seeds or fish.

MEIKLE'S APPARATUS FOR EXTRACTING BONES, SEEDS, FISH, ETC.

The cylindrical vessel A (Fig. 29) is divided into an upper and lower chamber by means of a perforated partition sup-

porting a layer of filtering material, and is also fitted with a hollow lid H, conical projections on the under side of which facilitate an evenly distributed dropping of the solvent. The hollow space within the lid can be heated or cooled by means of the contained steam and cold water pipes. The steaming chamber is steam jacketed and is also fitted with a small steam jacket or steam disc on the inside. The lower chamber is provided with a similar heating appliance, and both are fitted with revolving stirrers D, mounted on a hollow shaft, which extends through the partition, and is perforated with lateral apertures.

When the apparatus is to be used the raw material is placed in the upper chamber and impregnated with the solvent, which is forced in under pressure, through a supply pipe perforated to ensure uniform penetration. The resulting fatty solution then filters through the partition and heat is applied to the lower chamber, whereby the solvent is vaporised and ascends, not through the filter, but by the easier passage, *vis.*, the hollow shaft, and escapes through the lateral apertures thereof. The vapours are condensed by the cold water pipe in the hollow lid, and the conical projections present the advantage of preventing the condensed liquid from collecting at one place; instead it drops uniformly from all the points formed by these cones, and so impregnates the material evenly. The extraction, therefore, proceeds continuously, and the stirrers can be set in action when convenient. When the operation is completed the hollow lid is no longer cooled, but heated like the rest of the apparatus, and the vapours then pass to the condenser. As soon as the extracted oil or fat is removed, the apparatus can be filled with a fresh charge of material.

APPARATUS FOR EXTRACTING FAT AND GLUE.

E. J. Machalski of Brooklyn has constructed an apparatus for this purpose, the details of which are as follow: At the bottom of the vessel A destined for the reception of the materials to be treated is placed a steam coil B for heating same. Three pipes C debouch into the vessel, one of them conveying

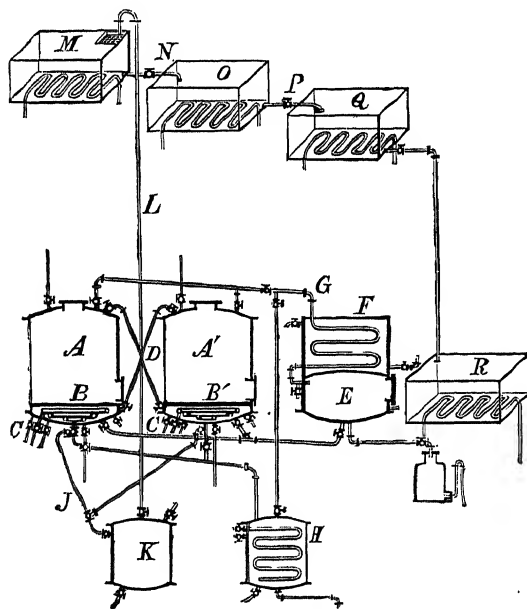


FIG. 30.—Machalski's extractor for fat and glue.

the (vaporised) solvent, the second removing the excess of this vapour to the condenser and the third supplying water. Underneath the vessel are placed recipients K, H for collecting the fat and glue, and there is also a battery of cisterns, M, O, Q, for concentrating and purifying the liquid glue, so arranged, one above the other, that the liquid runs through the series by gravitation, to be finally forced up through a pipe into special clarifying vessels.

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SCHWEITZER'S EXTRACTION PLANT

for animal (and vegetable) materials is composed of an extraction cylinder fitted with manholes at the top and side, a condenser, an evaporator, and the vessels destined to receive the condensed solvent. The pipes and valves effect the following combinations: Extractor with condenser, evaporator and condenser, condenser and storage vessel. The apparatus

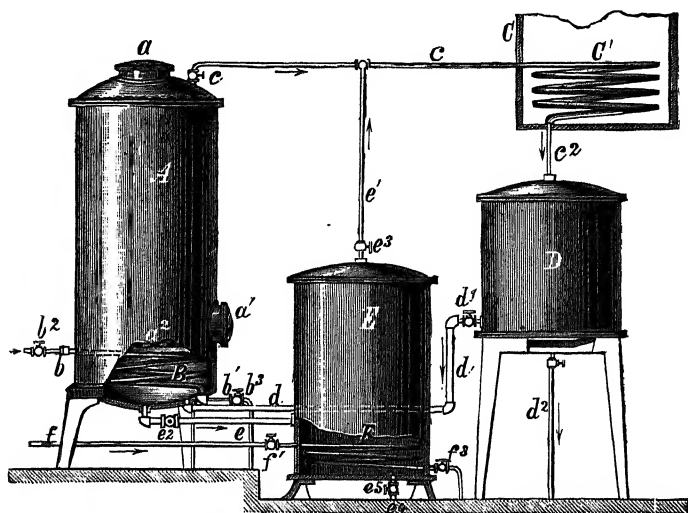


FIG. 31.—Schweitzer's extractor for animal material.

produces a perfectly dry residue, because the solvent used has a much higher boiling-point than that of water.

The method of working is as follows: The materials are placed on the grating in the extractor, and the solvent is admitted. Then all the communications are shut off except that between the extractor and condenser, and the solvent is heated by steam pipes nearly to boiling, whereby chiefly water vapour is evolved. The fat is dissolved and falls through the grating, and when the extraction is terminated the fatty mixture is transferred to the evaporator and heated to boiling,

whereupon the connection leading to the condenser is opened and the fat or oil afterwards drawn off. If the residue is to be freed from the small portion of adherent solvent it is then heated to boiling in the extractor, and the evolved vapour is led into the condenser.

W. O. Robbin's Extractor.—This apparatus (Fig. 32) is described by the inventor as consisting of a cylinder (for containing material to be extracted) traversed by a perforated tube for

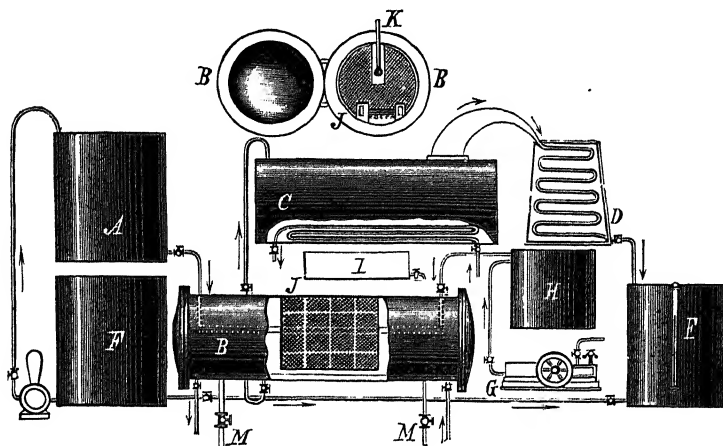


FIG. 32.—W. O. Robbin's extractor.

the admission under pressure of the solvent or of air. In the interior of the cylinder runs, on a guide way, an accurately fitting carriage, for the reception of the raw material, and provided with a slip extending from top to bottom. The operation is effected by filling the carriage with the material under treatment, the latter being then exposed to the action of the selected solvent, passed into the cylinder under pressure. When the extraction is completed the excess of vapour is driven over into the condenser by air, whereby the press residue is simultaneously dried, and the fat run off.

PERFECTED EXTRACTION APPARATUS.

In various other extraction processes the separation of the fat and the concentration of the crude glue liquor have to be effected in separate, independent vessels, an arrangement entailing special attendance on each. In the apparatus now to be described, however, the entire process, *viz.*, the steaming of the raw material, the recovery of the pure fat and the concentration of the glue liquor, is performed at one time and in the same apparatus. To this end the pipe *d*—as can be seen in Fig. 33—through which the liquid flows from the vessel A

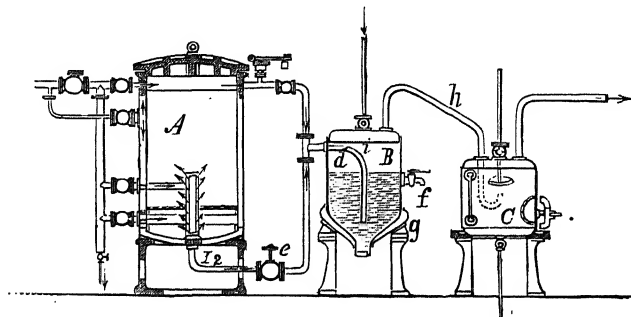


FIG. 33.—Apparatus for recovering fat and glue from animal residues.

to B, is provided with a fairly wide aperture *i* at the upper end, whilst the lower end reaches nearly down to the bottom of the vessel. On opening the valve *e*, the mixed liquid and vapour pass over from A into B, and the vapour is conducted away through *i* and the pipe *h* to the condenser C, whilst the liquid portion runs quietly down the pipe *b* and separates at the bottom of the vessel into fat and glue liquor, without, as hitherto, being mixed up together and heated by the current of steam.

The separation of the steam can also be effected in another way. Thus, for example, the pipe *d* is diverted sideways into a boiler, in the lid of which is placed a wide, open pipe,

whilst from the lower part of the pan the pipe *d* extends nearly to the bottom of the vessel. The mixture of steam and liquid flowing in tangentially through the pipe *d* streams along the walls of the pan whereby the steam is separated from the liquid, the latter flowing downwards through a pipe *d*, whilst the steam escapes through an upcast pipe *l* and passes thence through the pipe *h* to the condenser C. In the completed form a plate is provided, against which the mixture of steam and liquid, entering laterally through *d*, impinges, and is separated into these components, the liquid running down through a pipe, whilst the steam passes round the plate and escapes upwards through the aperture. The same separation can, of course, also be effected by other means. After the complete separation of the fat from the glue liquor has been accomplished in the lower part of the vessel B, the former is drawn off through the tap *f*, and the residual glue liquor concentrated in B itself. To this end a heating apparatus, *e.g.*, a steam jacket, is placed at the lower extremity of the vessel B, and by this means the glue liquor is raised to boiling-point and thoroughly concentrated. The vapours thereby expelled pass through the pipe *h* to the condenser C, where they are completely liquefied, the concentration being in this manner effected entirely without smell. The nitrogenous dry matter in the glue liquor is left as a residue and may be removed by a suitable opening provided in the vessel B.

This apparatus consequently offers the great advantage that the steaming process in A is in nowise influenced by the concentration in B, and both operations can be carried on at the same time; only for that purpose the condenser must be of sufficient capacity to cope simultaneously with the vapours delivered by A and those evolved from the glue liquor in B.

Whereas, in earlier forms of apparatus, the products consisted of dried bones (or meat), fat and glue liquor, in the

present instance they comprise merely fat and the whole of the solid substances present in the carcase. By the abolition of the easily decomposable glue liquor, an important hygienic advantage is, however, gained without the other favourable points of the process—especially as regards absence of smell—being impaired.

The quality of the solvent used is not without influence on the quality of the fat produced by any of these processes. The defects in the use of carbon tetrachloride (p. 61) have already been noted. Benzine or benzoline or petroleum spirit is the solvent almost universally used because it is cheap, although its inflammability is a great bar to its employment. Some samples are of good quality and evaporate away without leaving any residue behind, but others contain a small quantity of a heavy petroleum oil which, at the temperature used in the extractor, does not volatilise and is left behind in the fat, imparting to it an undesirable petroleum odour. This difficulty can be avoided by paying particular regard to the quality of the benzine used, or by subjecting the solvent to a preliminary distillation in the apparatus itself and rejecting any portion which does not volatilise at the temperature employed.

The extractor made by Engel Extractors, Ltd., is shown in Fig. 34.

The Engel extractor consists of the following parts :

1. The extractor.
2. Distiller.
3. Vapour condenser.
4. Solvent tank.
5. Preheater.
6. Separator.
7. Vacuum discharger.

and the manner of working is as follows :

The material to be treated is charged into the filling hole
A. Solvent is then run in from the solvent tank B, passing

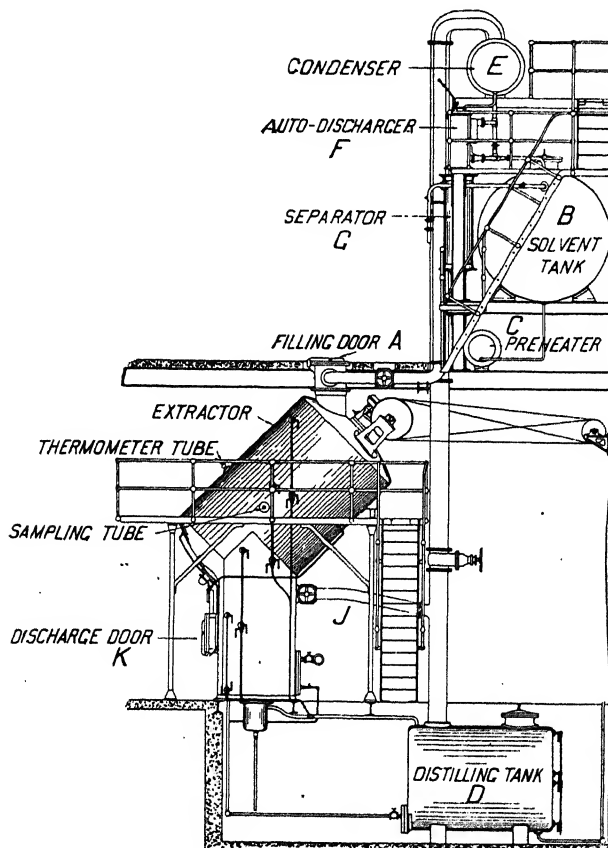


FIG. 34.—Engel extractor.

on its way through the preheater C, which raises it to the required temperature.

Steam is then admitted to the heating coils in the base of the extractor, and the material is continuously agitated until the extraction is complete. The solution is then discharged

to the distiller D, where the solvent is evaporated off and goes to the condenser E, and from there through the vacuum discharger F and separator G to the solvent tank B, the extracted oil being pumped to the oil storage tank. The residue is discharged through door K.

To economise heat, the volatilised solvent from the distiller can be returned to the meal through a bye-pass pipe J, and in cases where a battery of two, three, or more extractors are operated in series, the solvent charges are used consecutively in rotation for extraction, and a common preheater, still, condenser, and generic equipment, is only necessary for the set of pots employed.

PRESSES.

Presses play a merely subordinate part in the recovery of animal fats and oils, whilst in the case of vegetable substances of this nature they form the chief mechanical appliances used. For animal fats they are employed in the following instances:—

1. For expressing the residue from the fat-rendering process (greaves: see Greave presses);
2. For pressing tallow to separate it into its solid and liquid components;
3. For pressing the blubber from marine animals.

Greave presses are simple spindle presses, but for the other purposes mentioned, hydraulic presses are mostly used, and these will now be generally described.

The hydraulic press is founded on the principle of the uniform transmission of pressure throughout liquids in closed vessels, and such presses consist of two separate parts—the press proper and the pump. The first is composed of a press plate terminating below in a piston (moving in the movable cylinder contained in the lower enclosed portion of the machine); a head plate; and (as a rule) four pillars connecting the press head with the base of the apparatus. In this latter

is also placed the connecting piece effecting a junction between the press cylinder and the pump cylinder. The pump is of the plunger type, with two cylinders of unequal diameter, the larger becoming automatically disconnected when the pressure attains about 20 in., the further pressure required being effected by the smaller cylinder alone. The minimum of pressure varies for each press, and the overstepping of the maximum pressure is prevented by the automatic action of a safety valve. The pressure produced by the pump is transmitted through the liquid—water, glycerine, or a mixture of both—and also through the connecting valve and the liquid in the press cylinder, and exerts on the piston of the latter a force per unit of space equivalent to the pressure per unit produced by the pump (small) piston. As soon as the desired pressure is attained the connecting valve is closed, so that the pressure in the press cylinder may not be influenced by any leakage in the pump. In order to reduce the pressure a second valve in the connecting gland is opened and the liquid returned to the pump chest without passing through the cylinder. In this manner the valve of the pump is preserved on the one hand, and on the other the same liquid is used over and over again, no renewal beyond the small amount wasted by leakage being required.

Hydraulic presses of both vertical and horizontal form are employed, the former being, however, the kind mostly used in tallow-melting works. In those employed for pressing fat, a number of press plates of trough or box form are superimposed, so that the lower part of each engages in the upper part of the one next below and presses the material contained therein, the outflowing fat being conducted into a common channel. For warm pressing, the press plates are fitted with appliances for steam heating, swivel pipes permitting the introduction of steam at one side of the cast- or wrought-iron plates and the removal of the condensed water

on the other. Where several hydraulic presses are at work in the same room, they can be all fed from the same pump; but where they are in separate rooms, then working by accumulators can be practised with great advantage.

A hydraulic tub press is shown in front and side view and in section in Figs. 35 and 36. The press consists of the press

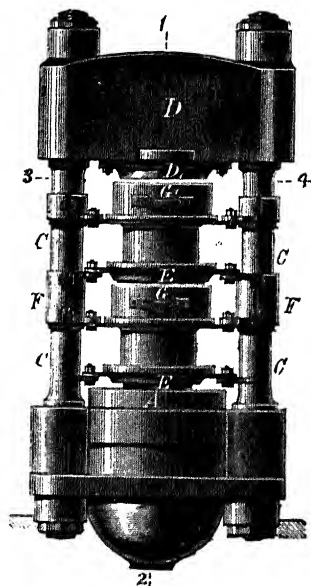


FIG. 35.

(Front view.)

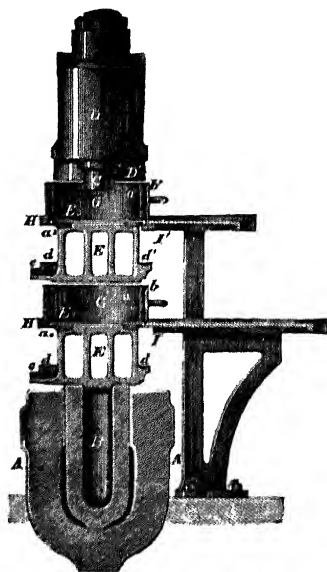


FIG. 36.

Hydraulic tub press.

(Side view and section.)

cylinder A and the piston B, situated between two pillars on which the troughs E slide by means of rings. These troughs carry a solid iron plate and are surrounded below by a channel *dd* for collecting the expressed fat. The boxes G, containing the fat to be treated, have double walls and are provided with a circular row of apertures at the top of the inner wall. The boxes are filled with fat, which may have

been previously cast in moulds of suitable size, and are then covered over with a horse-hair cloth. When the press is started the troughs E are pressed into boxes G, the liquid fat runs out of the openings below the upper rim of the latter and collects in the channels *d*. As soon as the operation is completed the pressure is removed, whereupon all the parts of the apparatus recede into the positions they occupy in the drawing. The boxes G are taken out on the table and replaced by others ready filled with fat, so that the time the press is standing idle is reduced to a minimum.

Among the new forms of hydraulic presses may be mentioned those of Brinck & Hübner, by reason of their several advantages, consisting of:—

1. Quicker and simpler service with greater capacity;
2. Abolition of expensive press cloths;
3. Production of uniform cakes of fat;
4. Great durability without attrition of expensive parts.

The general construction is as follows: In the press are situated four, six, eight or ten wrought-iron or steel rings, one above another, and each fitted with a finely perforated steel bottom. Between each pair of rings is placed a cast-iron or cast-steel press plate, convex on the upper surface but flat on the under face. On these plates, which are guided by the pillars of the press, rails are affixed for carrying the press rings, and on these the latter slide when being placed in, or removed from, the press; and, in addition, each press plate is provided with a large encircling channel to catch the expressed liquid fat. The presses are filled by simply laying over the perforated steel bottom of each ring a horse-hair, wool or felt cloth, covering this with the fat to be pressed, and the latter, again, with a second similar cloth. When all the rings are full the pressure is turned on and the fluted upper face of each plate forces its way up into the ring next above, the liquid fat running out through the

horse-hair cloth, the perforated steel plate and the grooves in the press plate, into the channel.

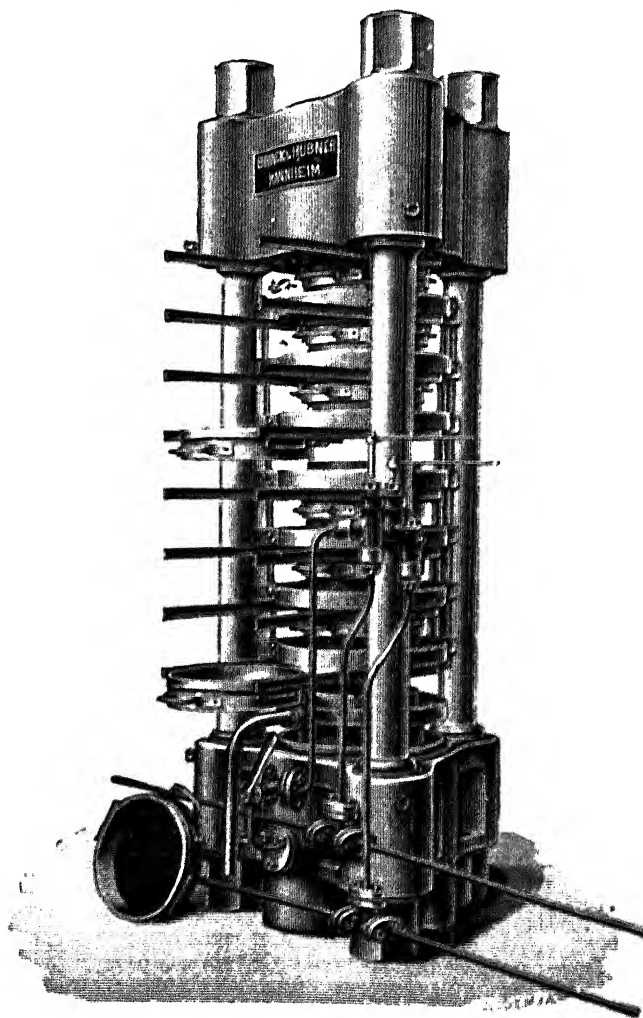


FIG. 37.—Brinck & Hubner's hydraulic ring press.

When the fat is caused to flow uniformly from the entire surface of the cake it has only a thin layer to traverse, and

thus runs away rapidly and yields a thoroughly uniform press cake. In pressing particularly heavy cakes the rings become

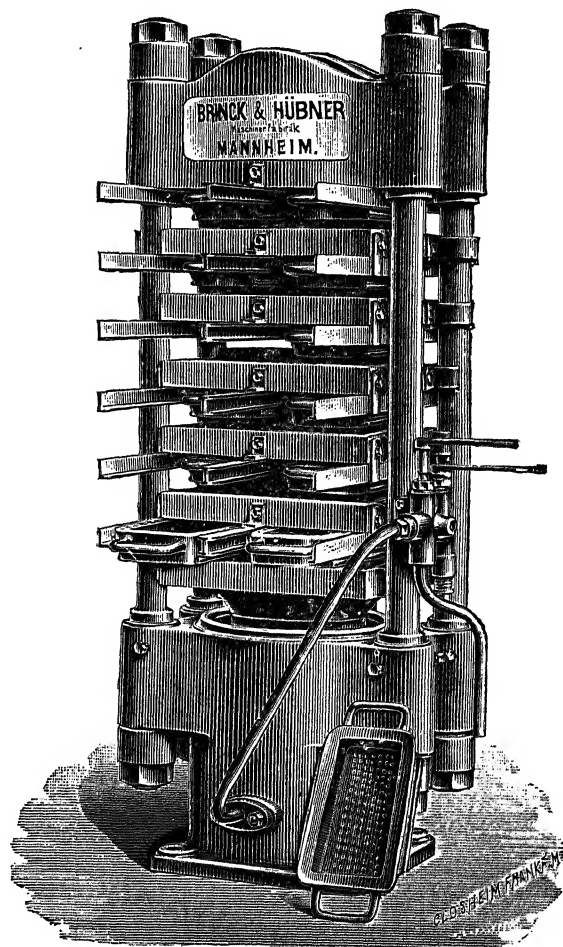


FIG. 38.—Brinck & Hübner's hydraulic box press.

too heavy to be lifted out, so on this account the presses are fitted with two lateral frameworks and a double set of rings, and as soon as one set of rings containing the pressed

fat is drawn out on to the side frame to be emptied and refilled, the other (filled) set is pushed into position from the other side frame, so that the operation is simple and goes on without interruption.

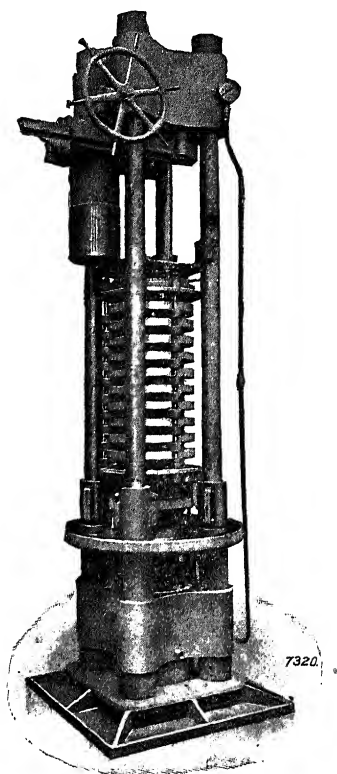


FIG. 39.—Manlove & Alliott's cage press system.

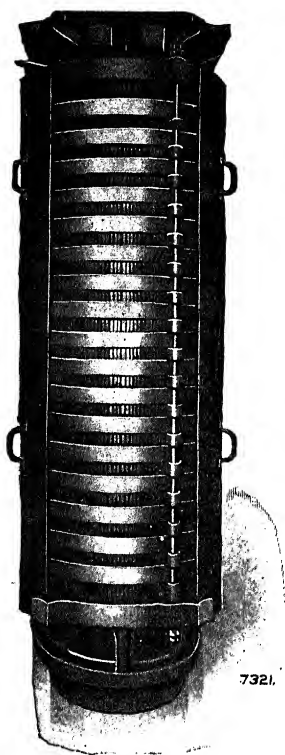


FIG. 40.—Cage of Manlove & Alliott's press.

By using suitable boxes in place of the rings the cakes can be made in square or trapezoid form of suitable size. All presses are provided with appliances for heating each part with steam or hot water, and, as each mould rests between two heated plates, the temperature is uniform, and results are

attained that no other system of pressing can yield. The presses are constructed for pressures of 300 atmospheres over, and the cylinders, pillars and nuts are of cast steel.

The cage presses manufactured by Manlove, Alliott & Co. Ltd., are suitable for the preparation of premier-jute animal fats.

These presses are constructed with a pressing cage in which the fat is placed either in bulk, or separated into plates, and can be treated either hot or cold (Fig. 39).

This pressing cage (Fig. 40) is constructed of bars arranged vertically, and supported by steel rings, the bars being closely arranged as to prevent any escape of the fat under pressure, whilst allowing free exudation for the oil.

The pressure is equalised through the entire material being pressed, by applying top pressure either with top or bottom loose head, which is withdrawn when charging the cage.

The pressing cage rises on to the loose head by means of side friction, but special provision is made whereby the cage is firmly held whilst this friction is being overcome, preventing the pressed material from falling.

These presses can be made for either hot or cold pressing and are obtainable in a variety of sizes, a maximum of 3 tons per square inch on the material under pressure being possible.

The presses illustrated (Fig. 41) are for the production of stearine by the cold process. They consist of hydraulic presses with ram 12 in. diameter, working pressure 2 tons per square inch. Each press is fitted with two carriages with wheels on rails, allowing for one carriage to be charged and in position in the press, where it is raised by means of the hydraulic ram and placed under pressure whilst the other carriage is being loaded in readiness for a further charge.

Each carriage is fitted with guides and guide bars.

plates. These latter pass through the press head when the carriage is in position in the press and thus prevent the plates being moved out of position due to side pressure.

When it is a question of pressing fat with heat, simple presses without boxes, rings or tubs are used.

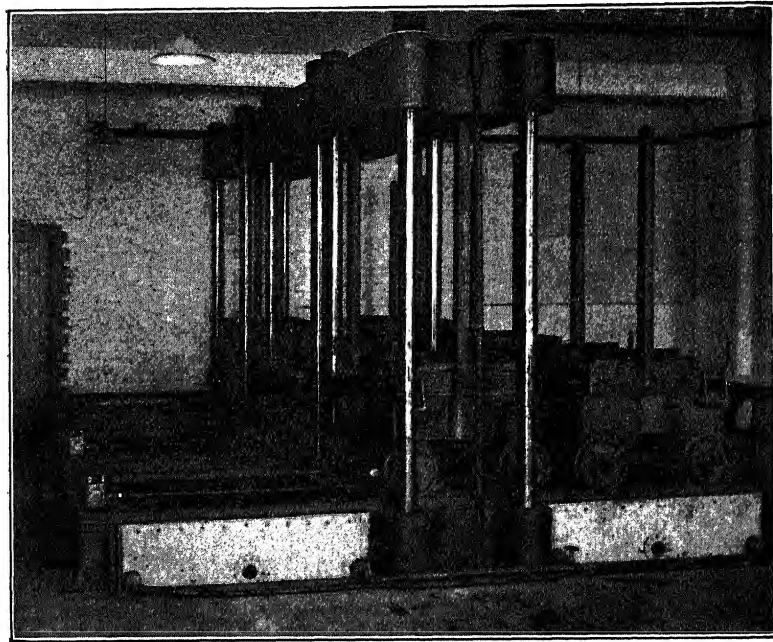


FIG. 41.—Stearine press (Manlove, Alliott & Co.).

The fat is packed in sacks and trimmed on the plate of the press, and when the loading is completed pressure is applied, whereupon the fat runs out and is caught in a suitable manner. The pressing of blubber is generally effected in very primitive presses worked by levers. Of course, a quantity of the product is lost, but Rose, Downs & Thomson of Hull have constructed a screw press for fish

oil highly suitable for this purpose (Fig. 42), only moderate pressure being required. The shaft carrying the screw spindle is fitted at either end with fast and loose driving pulleys, that on the end being driven by a straight and the other by a crossed belt, so that the plates of the press can be raised or lowered by steam power, and also, when necessary, stopped at once. The actual press is placed within a

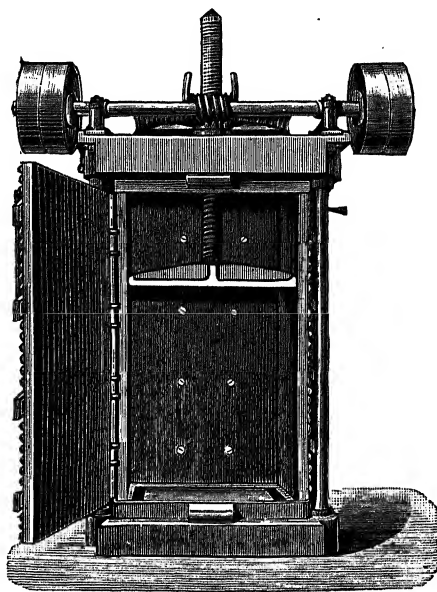


FIG. 42.—Fish oil screw press (Rose, Downs & Thompson).

steam-jacketed chamber, the outer walls being of wrought-iron strong enough to withstand the pressure, whilst a door of similar construction and also heated by steam forms a secure and hermetical seal during the time the press is at work. The charge is packed in sacks which are laid between metal press plates. The press can also be constructed to work with hydraulic pressure, while for certain purposes the jacketing chamber is omitted.

FILTERING APPARATUS.

The animal fats do not require to be put through such a complete system of purification as vegetable products of the same class. In the first place, the animal fats are generally solid and therefore have to be brought into the liquid condition before they can be purified or filtered; on the other hand, they are mostly put to such technical uses, that they sustain a certain amount of alteration, *e.g.*, saponification or

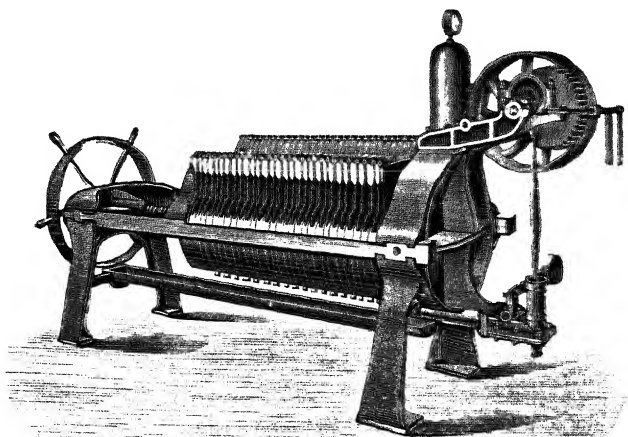


FIG. 43.—Filter press worked by power (Rose, Downs & Thomson).

melting, which purifies them. The fats destined for alimental purposes are prepared by graduated or fractional pressing, then again they differ from the vegetable fats in that very little of the solid and extraneous matters escape through the press cloths, so that further purification is usually superfluous.

As a matter of fact, it is only the liquid fats of the animal kingdom, the fish oils, that are purified by filtration when required for alimental or high class technical purposes. The reader is therefore referred for information on the subject of

filtering apparatus to the author's work on *Vegetable Fats and Oils*, where they are more fully described.

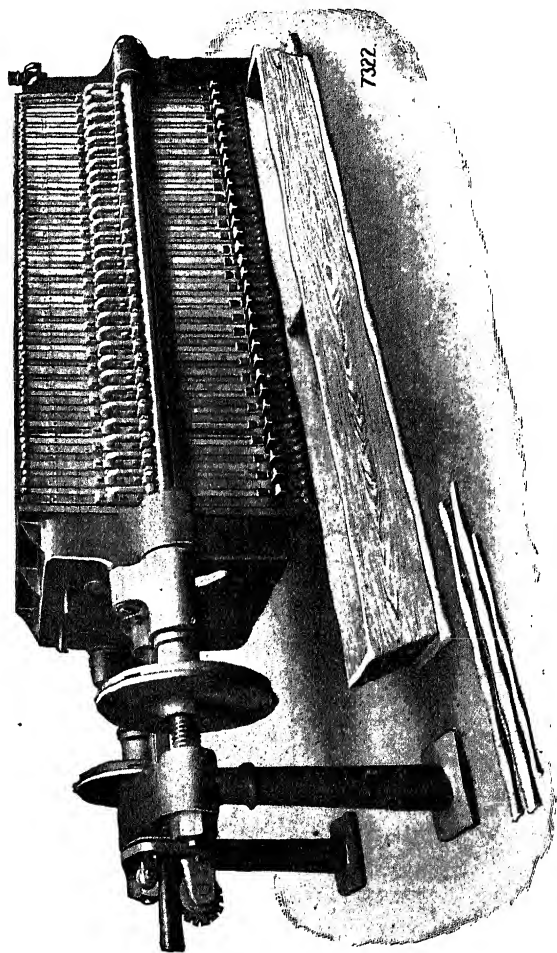


FIG. 44.—Manlove & Alliott's filter press with wood plates.

FILTER PRESS FOR LIQUID ANIMAL FATS (FISH OILS).

Fig. 43 depicts a filter press particularly suitable for filtering fish oils, and capable of treating twenty tons of oil

with ease; moreover, the press can be worked every day for a week at a time without requiring cleaning. It has thirty-six press plates, and as soon as the separate divisions have

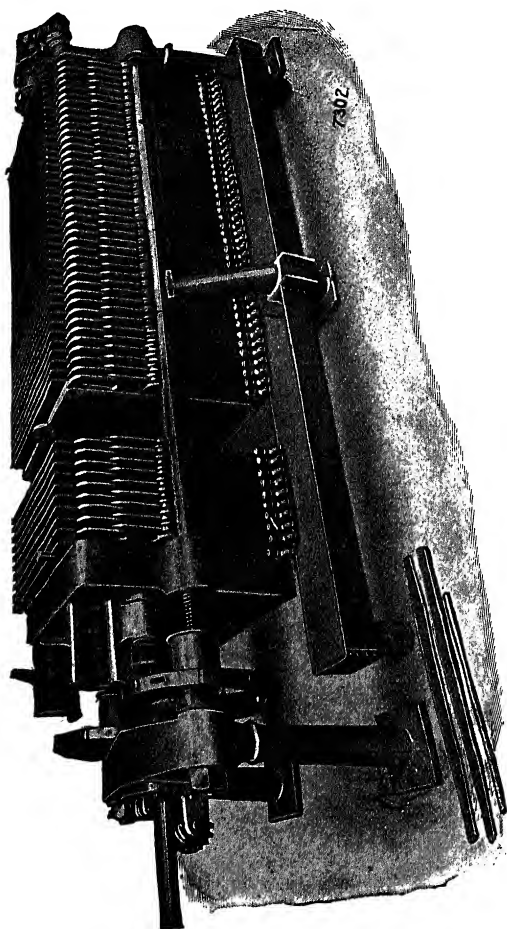


FIG. 45.—Manlove & Alliott's filter press with iron plates.

been fitted with the filter cloths and the screw tightened up the machine is ready for work. The material to be filtered can be fed direct and the filtrate removed by pumping. The

press is self-contained, contains no setting and can be easily fitted up, all that is required to make it ready for work being connection with a source of power, direct or through intermediate shafting.

Figs. 44 and 45 are also types of presses suitable for the filtration of fish oils.



CHAPTER IV.

ANIMAL FATS AND OILS: RAW MATERIALS, PREPARATION, PROPERTIES AND USES.

EDIBLE ANIMAL FATS.

BUT few of the animal fats are used for edible purposes ; butter, lard and dripping almost exhaust the list. The most important is

BUTTER.

Raw Material.—Cows' milk, as being the most abundant of the secretions of all mammals.

Preparation.—Milk contains a fat—butter—which is agglomerated when the milk is exposed to strong and continuous agitation. The phenomena have been explained by Soxhlet as follows: The fat forms, along with the other constituents of the milk, a perfect emulsion of small spherules of very different sizes. In this condition the fat is endowed with the property of remaining liquid at temperatures whereat the butter obtained from the same would be solid. This peculiarity results from the surface tension proceeding from the extremely thin layer of serum (also known as the serum envelope) lying within the sphere of attraction of the globules. In the same way as molten masses in general can be made to congeal by vibration, so in the manufacture of butter a few drops are first made to solidify by mechanical concussion and these increase in size by contact with the liquid fat. The larger congealed particles of fat thus produced attach themselves by impact to others, and so, finally, the greater part of the fat

becomes collected into small mulberry-like adherent masses separable from the other constituents of the milk by skimming. Therefore, in the process of butter-making, the fat globules, on the one hand, are congealed, and on the other, the solidified particles are united to form a coherent mass.

In chemical composition milk is a highly complex substance. The following constituents occur (though in quantities varying between certain limits) in all sound milk: casein, albumin, fat (butter), lactose (milk-sugar), salts and water. The proportions vary, even in one and the same animal, according to the mode of feeding, time of year, age of the individual, etc., within certain limits, so that a universally applicable analysis of milk cannot be given. The appended figures are, however, the mean of a large number of analyses:—

Cow's Milk.	Average Percentage.	Limits of Variation.
Water	87.5	83.0-90.0 per cent.
Solid (dry) matter	12.5	10.0-17.0 „
Containing:—		
Fat (butter)	3.4	0.8-8.0 „
Casein	3.2	2.0-4.5 „
Albumin	0.6	0.2-0.8 „
Lacto-protein	0.1	0.08-0.35 „
Milk-sugar	4.5	0.3-6.0 „
Ash	0.7	0.7-0.9 „

The following analyses show the composition of whole and skim milk:—

	Whole Milk.	Skim Milk.
Fat	3.50	0.46
Casein and albumin	4.75	3.37
Milk-sugar (lactose)	4.17	5.34
Ash	0.70	0.72
Water	86.88	90.11
	<u>100.00</u>	<u>100.00</u>

The greatest care and cleanliness are essential in the preparation of butter, by reason of its extreme liability to absorb extraneous flavours and odours, the best fresh milk immediately

undergoing an unfavourable alteration under the influence of even very small quantities of fermented or sour milk. For this reason storage chambers for milk have to be selected with great care, and attention devoted to keeping them scrupulously clean.

Very widely divergent methods are employed for preparing butter from milk. In all the milk is divided into two portions:—

1. A cream very rich in fat, this being taken for churning ; whilst
2. The second portion (poor in fat), the skimmed, blue thin milk, is put to another use.

In separating the cream from the milk three different methods are practised :

1. The milk is left to itself as a layer, some 4 in. deep, in shallow pans (shallow setting) until the cream collects as a consistent mass on the surface.
2. The milk is set in high vessels, 16-20 in. in height (deep setting, or Schwartz process), and kept at a temperature of 2°-4° C.
3. The milk is separated almost instantaneously in special centrifugal machines (milk separators) into separated milk and cream.

The first method is the oldest, and is still practised in many dairies. The vessels employed are either of wood, well-fired stoneware, enamelled cast-iron or pressed tinplate. When the cream has finished rising it is skimmed off with a flat spoon or separated from the skim milk by means of special appliances. In small dairies basins are often used from which the skim milk can be poured away direct, the cream being retained by a cream catcher. When kept for some time, or when the cream is a long while in rising, the products—skim milk and cream—arrive at the initial stages of acid fermentation.

With regard to No. 2, this method presents the advantages of keeping the products sweet, requiring fewer vessels, occupying less room, and being independent of the state of the weather. On the other hand, the cream is thinner and more bulky; consequently the yield of butter milk is increased.

No. 3. In the process of "separating" milk, the liquid, enclosed in a rapidly revolving drum, is, by the action of centrifugal force, affected in such a manner that the specifically

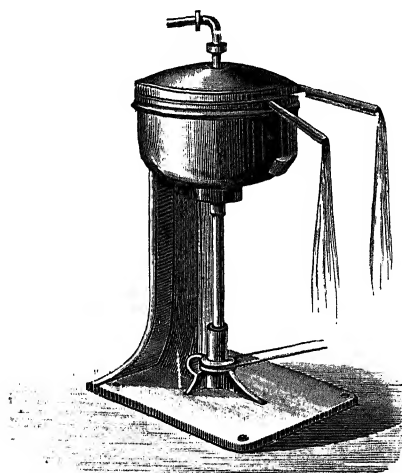


FIG. 46.—The De Laval separator.
(Elevation.)

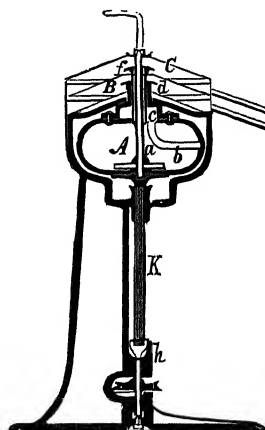


FIG. 47.—The De Laval separator.
(Section.)

heavier milk is impelled against the outer walls, whilst the specifically lighter cream is forced upwards and towards the centre. This process enables the work to be carried on uninterruptedly, producing a constant flow of sweet cream and "separated" milk. Among the various types of cream separators that of De Laval (Fig. 46) has proved the best in practice. In this apparatus the fresh milk is run in continuously, whilst the cream and the skim milk leave the machine by two separate outlets, these products being obtained in such a condition that the cream may be either sold as a luxury

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of diet or worked up into butter, whilst the residual milk makes an excellent cheap food. The separator consists of an oval hollow vessel A (Fig. 47) of best Bessemer cast-steel, and holding about $1\frac{1}{2}$ galls., which is encased in a cast-iron bowl and rests on the spindle K. This latter stands loosely in a wood block bearing in a depression in the pivot $\frac{1}{2}$, which is driven by the small band pulley and intermediate gear at such a rate that it makes 6000 revolutions a minute, carrying the spindle and the receiver with it. The central supply pipe a , with two lateral delivery tubes, is screwed in the bottom of A, and over this is slipped the tube f leaving an annular space of about $\frac{1}{4}$ in., the lower end of which is fastened to a ring which forms the bottom of the small chamber c ; and over f again is placed the tube d , which expands below into a cup-shaped cover forming the upper part of the chamber c , the whole being joined to A by four screws. From the bottom of c projects the bent open tube b , reaching to the inner periphery of A. If now the milk be admitted through a into the receiver, which is running at full speed, it is forced with great power against the walls, the time required to fill A sufficing for the separation of the milk into its constituents, of which the specifically lighter cream collects around the central supply pipe and is driven upwards as the supply proceeds into the narrow annular space between a and f and to the chamber c , whence it runs away through a pipe. The thin milk rises up through b into the small chamber c and thence through the opening between d and f into the chamber B, whence it also escapes through a discharge pipe.

The Bechtolsheim "Alpha" separator, driven by power, will treat up to 460 galls. of milk per hour, leaving only about 0.2 to 0.3 per cent. of fat in the separated milk; smaller machines are also made for hand driving, to separate 15 to 60 galls. per hour.

In Fesca's centrifugal machine (Fig. 48) the operation is

discontinuous. The milk enters through the funnel *a* into a roomy cylinder *A* running at high speed, whereby the cream is forced, in the direction indicated by the arrow, towards the centre, where it is retained by the cylinder *b*, whilst the thin milk runs away from the periphery of *A* through openings in the bottom, and, entering the jacket space *B*, is run off through the pipe *C*. At the end of about an hour the liquid escaping from *C* is not merely thin milk, but milk that is only slightly

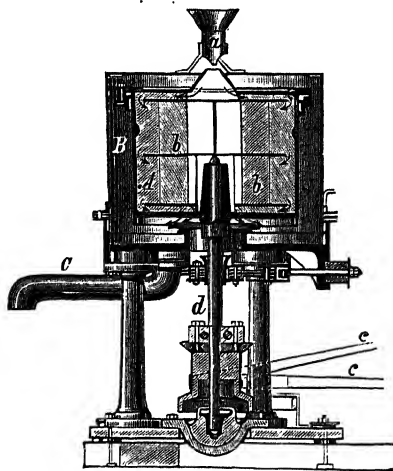


FIG. 48.—Fesca's centrifuge.

separated, owing to the chamber *b* being full of cream. When this is observed the driving belt *c* is slipped on to the loose pulley and the drum is allowed to empty itself. As the centrifugal force decreases the cream runs out into the jacket space and is caught in a separate vessel at *C*. When it is all out the operation can be recommenced.

The separation of the cream is effected more readily at somewhat higher temperatures than it is in the cold, so that it is advisable either to put the milk through directly it comes from the cow or else to warm it up to blood heat. On the

other hand, the keeping qualities of the butter improve in proportion as the milk after milking, or the cream after separating, is brought down to the desired low temperature, for which purpose special coolers are used, consisting of a cooling chamber for the liquid, surrounded by one or two series of tubes through which cold water is continuously flowing, the product being drawn off after a certain time into a vessel beneath.

As we have already seen, sweet or sour cream is obtained as the raw material for butter-making, according to the system pursued. No difference is observed in the yield of butter whether the cream be sweet or sour, but there is a distinct difference in flavour, sweet cream yielding a sweet, nutty-flavoured butter, whilst that from sour cream has a sour and (when the cream has been kept long) even a rancid taste. The temperature of the cream for butter-making should be about 15°C . in summer and 20°C . in winter. The principle of the process has already been discussed. It is necessary to subject the cream to powerful and uniform agitation, the regulation of which constitutes a very important part of the process. For this purpose a large number of appliances have been devised, but it is impossible to refer to them all.

The types of construction may be divided into classes, according to their action, *i.e.* :—

1. Beating (dashing),
2. Stirring,
3. Shaking.

As to which form of apparatus is the best the opinions of butter-makers widely diverge. So far as the constitution of the butter itself is concerned, it is immaterial which method is adopted, the only conditions to be fulfilled being that of forming the butter in the shortest possible time and in the highest degree of purity.

Among the appliances may be mentioned : The percussion

churn (farmer's churn), which is the oldest but least recommendable, by reason of the great expenditure of force required; Lefeldt's rotary barrel churn; Davis' rocking churn; the roller churn; the Holstein barrel churn, etc.¹ Among those suitable for work on a large scale Rennes' butter machine (Fig. 49) should be mentioned: this belongs to the type of percussion and dasher churns.

The milk is placed in a box J so arranged that there is no

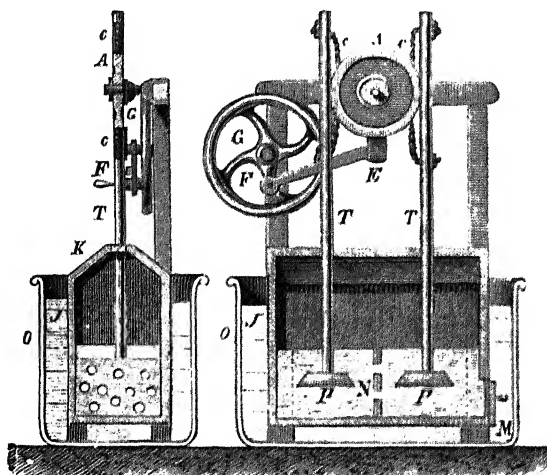


FIG. 49.—Rennes' butter machine.

difficulty in running in the cream and removing the butter, both being effected through the flap door K. The buttermilk is drawn off through an opening at the bottom of the vessel, generally kept closed by means of a ball valve. The beating of the milk is accomplished by a pair of dashers P P alternately raised and lowered by eccentric gearing. A disc A with grooved rim rotates on a fixed axis and carries the two ropes c c, which are also connected with the rods T T attached to

¹ *Translator's Note.*—There are many makers of these different styles of churns, those above being evidently intended by the author as typical examples.

the dashers P P. A also carries an iron arm connected with the fly-wheel G at F by a rod E, the pin F forming at the same time the crank of the fly-wheel. The distance from the centre of the guide disc to the point of attachment of the connecting rod with the iron arm being greater than the radius of F, A can therefore merely rotate through an arc the radius of which is double that of F, while F makes a complete turn. The arm is rigidly attached to the disc and therefore the latter can only make an alternating semicircular movement, during which the points at which the ropes *cc* are attached have a similar motion, *i.e.*, a semicircle, whereby the attached rods T T are caused to rise and fall alternately. These rods move vertically through suitable apertures in the cover of the box J, and since the ropes *cc* are placed exactly opposite one another the one rod T begins to rise at the same instant that the other rod commences its descent. A perforated partition N is placed between the dashers P P, and, finally, the cream chamber J is surrounded by a vat O which can be filled with hot or cold water as desired.

Brochardt's butter machine (Fig. 50) belongs to the class wherein the separation of the butter is effected by the stirring produced by the vertical rotation of a shaft. This machine consists of driving gear K, which, by means of wheels I and J, sets a shaft B in rapid motion. B carries a series of vertical dashers and also a tube *d* connected with a toothed wheel H, which in turn is in connection with the wheel I. At the lower end of the tube *d* are fixed a number of horizontal rods F, forming the spokes of a wheel, while a similar wheel is situated below, being connected with the upper one by the vertical dashers G. The shaft *c*, which is attached to the box of the lower wheel, is supported in a bearing in the bottom of the churn. There are thus in the machine two systems of dashers (G, C), both of them movable, though in opposite directions. This opposite motion, produced by the force acting on the two

cone wheels H and I from the wheel J, causes, as can readily be understood, a very brisk movement of the cream in the churn, quickly effecting the separation of the butter.

The proposals which have been made for admitting air, or for adding acids or alkalis to the cream in the butter-making, in order to facilitate the rapid separation of the fat, have proved unsuitable.

The butter produced by one or other of these mechanical

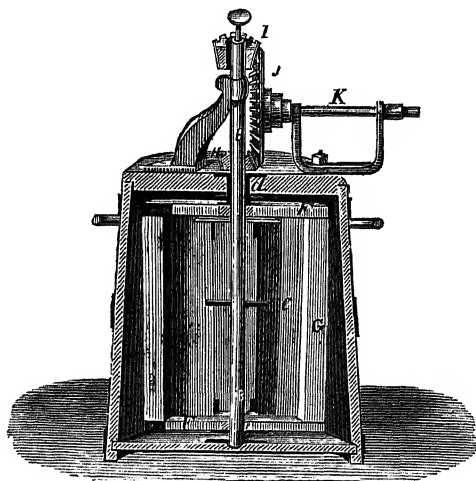


FIG. 50.—Brochard's butter machine.

processes contains a large proportion (16 to 22 per cent.) of enclosed buttermilk, which causes the butter to quickly become rancid, unless thoroughly expressed. This buttermilk contains both milk-sugar and casein in solution, when sweet cream has been used, or, when the cream is sour, solid casein, in addition to dissolved lactic acid. All these substances are readily decomposable, the milk-sugar being converted into lactic acid by the lactic ferments present, and then causing the casein to curdle. On this account the cavities in butter pre-

pared from sweet cream and not further purified very soon exhibit small lumps of curd together with a liquid tasting strongly of lactic acid.

After a short time, particularly in summer, the lactic acid is further converted into the evil-smelling butyric acid, whereby the butter assumes a repellent taste and smell. When the butter has been made from sour cream, then lactic acid will be present ready-formed in the butter, and the decomposition of this acid into butyric acid will occur much sooner than in the case of sweet-cream butter. The decomposition, once set up, extends also to the fat, the butter very soon assuming a disagreeable, irritating taste and cheesy smell, which render it unfit either for eating or cooking purposes. Butter which has undergone such alteration can be again rendered suitable for cooking only by a carefully performed re-melting.

It is, therefore, essential that the buttermilk should be removed in order that the butter may keep and taste well, and the more completely this is effected the more successfully will such object be attained. As demonstrated by experiments (particularly on a small scale) wherein the butter was carefully freed from the adherent buttermilk, really pure butter prepared in this way is surprisingly stable, when it is kept at a temperature not exceeding 15° - 16° C., the pure fat being then much less liable to decompose than when buttermilk is present. In order to remove the latter the butter must be carefully washed with cold water, care being taken at the same time that the butter is in as finely divided a condition as possible, and that all the particles thereof are brought into contact with the water which is continually removed.

Working the butter by hand is impracticable in large dairies, therefore special machines have been made in which the butter is worked, either between grooved rollers or on a table, by a kneading roller, driven by hand or power. As a rule, eight or ten kneadings are sufficient to force out all the

buttermilk, after which the butter is made up into the form in which it is to be sold.

In winter time, when the cows are fed largely on hay and root crops, the butter is very pale in colour; it is usually the practice to remedy this by using butter colouring. At present general use is made for this purpose of annatto butter yellow or, in small dairies, even carrot juice, the butter being either mixed or kneaded up with the colouring matter or the latter is added to the cream before churning, the latter plan resulting in a more uniform distribution of the colour.

Properties of Butter.—The colour of butter varies considerably, ranging from a very pale cream to a fine deep yellow, winter butter being generally the palest, whilst summer butter is more highly coloured, this characteristic greatly depending on the composition of the fodder. The consistency is to an uncommon degree influenced by the prevalent atmospheric temperature, the same butter being fluid in summer and hard and solid in winter. Taste and odour should be agreeable and purely butter-like.

Unmelted unsalted butter contains under normal conditions, according to König:—

Fat	87.0 per cent.
Casein	0.6 „
Milk-sugar	0.5 „
Salts	0.3 „
Water	11.7 „

The composition, however, varies considerably, in that on the one hand the fat may amount to 90 per cent., whilst on the other the water may form as much as 35 per cent.

In consequence of its content of water and casein, butter quickly turns rancid. Attempts are often made to combat this rancidity by kneading the butter with 3 or 4 per cent. of common salt after thorough washing. According to Benedikt the same result may be better attained by keeping the butter

in a melted condition until it has thoroughly clarified and then separating it from the precipitated water and casein. Milk fat treated in this way is, however, no longer saleable as butter, as it lacks the characteristic appearance and flavour of this article.

Apart from traces of colouring matter, lecithin, cholesterol, etc., pure butter fat consists solely of the tri-glycerides of the fatty acids. Up to the present, acetic, butyric, caproic, caprylic, capric, lauric, myristic, palmitic, stearic, arachic and oleic acids have been isolated therefrom. An unusually high percentage of glycerides of the volatile fatty acids is specially characteristic of butter, but the bulk is composed of palmitin, a little stearin (classed both together as margarin) and olein.

The composition of butter is:—

(According to Schaedler).

Margarin	66 per cent.
Olein	28 "
Butyrin	6 "

(According to Winter-Blyth).

Stearin and palmitin	50.0 per cent.
Olein	42.2 "
Butyrin	7.7 "
Caprin, caprylin	0.1 "

It may be calculated approximately as follows:—

When the Hehner number of a butter is found to be 87.5 and the average molecular weight of the insoluble fatty acids 270, then the sample contains 91.93 per cent. of glycerides of the non-volatile fatty acids—olein, palmitin and stearin.

If the iodine number be 30.1, the iodine value of olein being 86.20, it follows that the olein content is 34.92 per cent.; therefore such a butter—the glycerides of the volatile fatty acids being calculated by difference—contains—

Palmitin and stearin	57.01 per cent.
Olein	34.92 "
Butyrin, caproin, etc.	8.07 "

100.00 per cent.

An indication of the nature of the volatile fatty acids is afforded by the Reichert number. According to Meissl, the fatty acids distilled from 5 grms. of butter require, on an average, 28.48 c.c. of $\frac{1}{10}$ -normal alkali for their saturation; consequently, the fatty acids from 100 grms. of butter require 56.96 c.c. of $\frac{1}{1}$ -normal alkali or 3.19 per cent. KHO. This corresponds to 0.778 parts of glycerine radicle (C_3H_5), which, deducted from 8.07, leaves 7.29 parts as the volatile fatty acids obtainable from 100 parts of butter. Therefore, 7.29 parts of volatile fatty acids require 56.96 c.c. of normal alkali for their saturation, and consequently their average molecular weight is about 125. The molecular weight of butyric acid is, however, 88, that of caproic acid is 116, of caprylic acid 144, and of capric acid 172. Consequently, Winter-Blyth's statement that butter fat contains 7.7 per cent. of butyric acid and only 0.1 per cent. of glycerides of other volatile fatty acids requires correction, the percentage of caproin, caprylin and caprin being much too low.

These calculations must not, however, be taken as absolute, seeing that the mean molecular weights of the volatile fatty acids obtained by the Reichert-Meissl process vary very considerably, Juckenack and Pasternack finding values varying between 95.1 and 98.3, Arnold found 98, and Siegfeld, 98.4-103. These figures would indicate a much larger proportion of butyric acid than that derived from the above calculation.

Violette calculates from analyses the composition of high-class butter fats to be as follows:—

Butyric	6.09-6.94	per cent.
Caproin	3.70-4.16	„
Glycerides of solid volatile acids	2.96-3.22	„
Glycerides of non-volatile acids (palmitin, olein, stearin)	85.98-86.62	„

The constants of butter fat vary within somewhat wide limits as given below:—

Constants of Butter Fat.

Specific gravity at 15° C.	0.936-0.940
" " " 100° F.	0.911-0.913
Melting-point	28°-34°
Saponification value	203.3-233.4
Iodine value	26.0-50.3
Reichert value	12.5-15.2
Reichert-Meissl value	20.63-33.2
Refractive index at 25° C.	1.4590-1.4620
Hehner value	86.45-89.8

Constants of Fatty Acids of Butter.

Specific gravity at $\frac{37.75}{15.5}$ ° C.	.9075-.91357
Melting-point	38°-45°
Solidifying point	33°-38°
Neutralization value	210-220
Iodine value	28-31
Refractive index	1.437-1.439

As revealed by the microscope, fresh cow's butter consists of perfectly spherical transparent fat globules. Hassall found crystals in older butter, these being, according to Mylius, best detected when the fat is examined under the polariser with crossed Nicol prisms, the crystals alone being then illuminated, whilst the rest of the field is in darkness. On using also a selenite plate the crystals give a beautiful play of colours. This method of examination serves to detect margarine or melted butter (see p. 123).

On prolonged exposure to the air, butter assumes the smell of tallow and becomes white. The setting of butter after melting does not progress uniformly, but a kind of crystallisation occurs. The portions nearest to the walls of the vessel are the first to crystallise, and they differ in composition from those remaining longer liquid in the interior of the mass. Sometimes this separation proceeds so far that an oil ("butter oil") and a solid fat or "stearin" are obtained, which can also be separated by allowing the melted butter to set at 20° C. and then subjecting it to pressure.

ADULTERATIONS OF BUTTER.

The additions made to butter are of a very divergent nature.

The coarser forms of adulteration consist of clay, chalk, gypsum, starch, meal, potato starch, ground white cheese, etc. Borax, water-glass and alum are sometimes added to preserve the butter and increase the weight by attracting the largest possible amount of water.

To impart a yellow coloration, small quantities of annatto, saffron, curcuma, azo yellow, etc., are frequently added.

The most important adulteration, however, is that of added extraneous fats, such as lard, tallow, goose fat, cotton stearin, coco-nut oil and palm-nut oil, and particularly oleomargarine. Though the addition of matters not belonging to the fatty series can be easily determined both qualitatively and quantitatively, the detection of extraneous fatty matters in butter was long attended with great difficulty, until suitable analytical methods were elaborated by Kötstorfer, Hehner and Reichert. Of these processes, however, Reichert's (or one of its modifications) alone still gives reliable results, since, according to Moore, mixtures can be prepared from oleomargarine and coco-nut oil which give exactly the same Hehner and Kötstorfer numbers as pure butter. By means of the specific gravity determination, the refractive index, and the Reichert-Meißl number the purity of a butter fat can be decided satisfactorily in every case, except when coco-nut or palm-nut oil have been added. In such cases the Polenska value is to be relied upon for giving a sure indication.

BEEF LARD OR RE-MELTED BUTTER.

Butter, as is well known, easily turns rancid if not carefully stored, and such butter is, therefore, for culinary pur-

poses frequently converted by a careful re-melting into "beef lard". When butter is carefully melted without addition over a fire, a froth, mainly consisting of unremoved casein, collects on the surface of the liquid; but the solution of milk-sugar in water, present in the butter as a result of insufficient washing, is, however, not so easily removable, and remains in the butter. In practice it is generally considered sufficient to skim off the froth from the liquefied butter, so long as any forms, and to then fill the butter direct into wooden tubs, where it is left to set.

When the melting is carelessly performed and the temperature allowed to rise too high, this results directly in the development in the liquefied butter of an irritating, unpleasant taste, due to the formation of certain decomposition products. Therefore, as butter is an expensive product, it is advisable to proceed carefully in melting, so as to preserve the quality, the increased expenditure of labour being amply compensated for by the improved results.

The best plan is to place the butter to be treated in a shallow vessel of thin, well-tinned sheet-iron, this being set in another which is filled with water, and to which heat can be applied. Butter being, like all fats, a bad conductor of heat, melting would take a long time unless facilitated by stirring. So long as any of the butter remains unmelted the water in the outer vessel may be kept on the boil, but as soon as the whole is liquefied the temperature is lowered until the butter is just fluid, and in this state it is left to repose for several hours without stirring.

Whilst in this condition of repose the particles of casein rise to the surface completely, while the aqueous liquid admixed with the butter subsides to the bottom, leaving the butter clear and transparent. Skimming is now practised, the most suitable method being to remove the uppermost layer by a skimming spoon and pour it on to a cloth of close

texture spread upon a hair sieve, through which the fat passes, leaving behind the particles of casein. Only when the surface of the butter has been cleared of every particle of froth is the removal of the fat proceeded with, and is then continued until only about one inch depth of fat is left in the vessel, the residual portion being allowed to solidify, in which condition it can readily be separated from the underlying layer of water.

The product ("beef lard") thus obtained is a perfectly pure butter fat, without the slightest trace of rancid or empyreumatic flavour, and it is much better to prepare butter for cooking purposes in this way than to run the risk of its becoming rancid.

A small sample of the "beef lard" taken from the setting vessel should be of a pure yellow colour and very translucent, a condition which can be attained by allowing the butter to cool only very slowly, *i.e.*, at a suitable and gradually decreasing temperature, so that the fat can crystallise uniformly. Usually the colour of the product is somewhat duller than that of the original butter.

TESTING BUTTER.

The following methods for the testing of butter fat may be adopted:—

Specific Gravity of the Fat.—Bell determines this constant at 37.8° C. ($= 100^{\circ}$ F.), at which temperature the sp. gr. of butter is 0.911 to 0.913, and that of oleomargarine and other fats 0.9028 to 0.9046.

König performs the estimation at 100° C. and finds for pure butter 0.866 to 0.868, or for adulterated samples 0.859 to 0.865.

According to Adolf Mayer, who works with the ordinary areometer, at 100° C. (or, more correctly, at the temperature of boiling water), and thereby obtains figures of merely relative, not absolute, value, the state of the barometer must

be taken into account, since a difference of 2 mm. in the barometer reading causes the sp. gr. to vary by 0.0001, so that the frequently occurring differences of 40 mm. of pressure will cause a discrepancy of 0.002, whilst the total difference in sp. gr. between natural and artificial butters amounts to only 0.007.

Casamajor employs the Hager method. The fat is melted in a spoon and one drop is introduced into 56.5 per cent. alcohol, the sp. gr. of which is exactly midway between butter (0.926 corresponding to 53.7 per cent. alcohol) and oleomargarine (0.915 = 59.2 per cent. alcohol) at 15° C. Should an air bubble adhere to the drop of fat it is detached by the aid of blotting paper. If the drop does not sink a slight concussion will cause it to do so if heavier than the liquid. Butter sinks to the bottom, whilst oleomargarine floats. If a drop of the melted sample be poured on to the spirit, warmed to 30° C., it will set, if butter, whereas oleomargarine remains liquid, although both float. On reducing the temperature to 15° C. the latter fat solidifies as well, and butter sinks. A "butter" that will not sink in 55 per cent. alcohol is oleomargarine with at most a third of cow's butter.

According to Moore, adulteration of butter by a mixture of oleomargarine and coco-nut oil cannot be detected by this means, since the sp. gr. of the coco-nut oil (0.9167 at 37.7° C.) is high enough to raise the sp. gr. of the mixture to that of butter (0.911 at 37.7° C.).

Emulsifiability.—According to Adolf Mayer, butter exhibits a greater capacity for forming emulsions than any of the fats employed to adulterate it, because the latter have been melted in the purifying process. He therefore sought to establish on this basis a simple method of testing, which, however, cannot be regarded as perfectly reliable.

Solubility.—The following tests for butter are based on the different solubilities of butter and its adulterant fats:—

Hoorn dissolves 1 grm. of the sample in 7 c.c. of petroleum spirit and leaves the solution to stand a few hours in tightly closed vessels at 10°-15° C. Butter fat remains dissolved, whilst veal fat, tallow and hog's lard separate out.

Münzel dissolves 1 grm. of the fat in 12.5 grms. of absolute alcohol (sp. gr., 0.797) on the water bath, closing the tube with a cork pierced to allow the passage of a thermometer, which reaches down to the bottom of the tube. The latter is then removed from the water bath, dried rapidly and the temperature at which the fat begins to set noted. Münzel obtained the following results:—

Commencement of Solidification.

Pure butter	34° C.
" + 10 per cent. of horse fat	37° "
" " 20 " " " "	40° "
" " 30 " " " "	44° "
" " 10 " " tallow	40° "
" " 20 " " " "	43° "
" " 30 " " " "	46° "
" " 10 " " hog lard	38° "
" " 20 " " " "	41° "
" " 30 " " " "	43° "
Margarine butter	56° "
Butter + 25 per cent. of oleomargarine	40° "
" " 50 " " " "	48° "

Horsley, Balland, Husson and Filsinger have made use of the varying solubility of pure and adulterated butter in ether or ethyl alcohol as a test of purity. Crook employed carbolic acid as solvent, and his results were confirmed by Lenz, with only a slight divergence in the values. Ten grains (0.648 grm.) of the filtered fat are dissolved in a gauged testing cylinder at about 66° C., shaken up with 1.5 c.c. of liquid carbolic acid (containing 373 grms. of crystallised carbolic acid and 56.7 grms. of water) and warmed on the water bath until transparent. After standing a while at ordinary temperature, one has either a clear solution (butter) or two

strata separated by a clear line of demarcation (beef, mutton, hog fat). The volume of the lower stratum amounts to:—

	Crook.	Lenz.
Beef fat	49.7 per cent.	— per cent.
Mutton fat	44.0 „	39.1 „
Hog fat	49.6 „	37.0 „

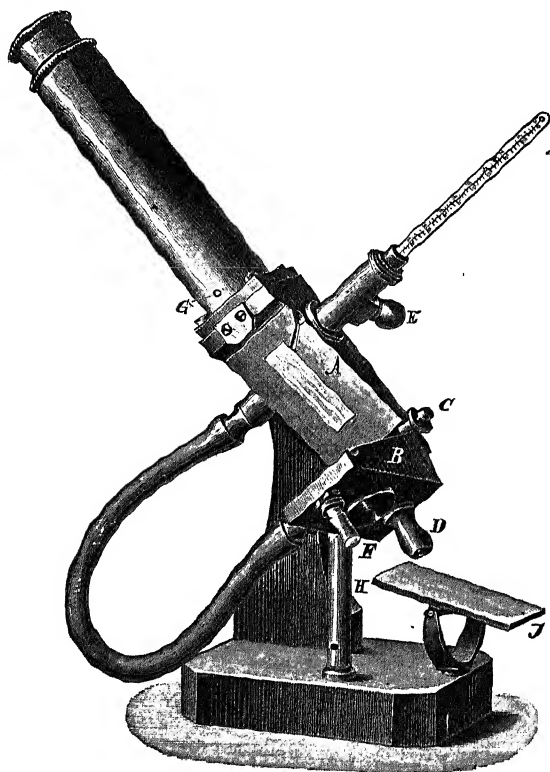


FIG. 51.—Zeiss' butyro-refractometer.

After sufficient cooling, more or less deposit is apparent in the upper layer. With 5 per cent. of hog fat Lenz could no longer detect any separation into two layers, but at the end of twenty-four hours a crystalline turbidity, differing from that observed in true butter, made its appearance.

More recently Zeiss' butyro-refractometer has been largely

used for butter testing, adulterations being rapidly detectable therewith. This instrument, supplied by C. Zeiss of Jena, is shown in Fig. 51. A and B are the hot-water jackets surrounding the two prisms and facilitating the testing of the fats at temperatures above their melting-points. C is a hinge on which B turns, D the water feed pipe and E the outflow pipe. The bayonet catch for the hot-water jacket is at F, and G is a pin serving to adjust the object; H serves as a support for A and B, and J is the reflector.

This instrument is based on the principle that the refractive powers of the fatty acids afford a means of determining their purity. It consists chiefly of a pair of prisms enclosed in a jacketed metal case, which, by the aid of a current of water, enables the substance enclosed between the two prisms to be kept at a constant temperature. Above the prisms is mounted a telescope with a scale, on which the position of the critical line of the substance between the prisms can be read off direct. According to the position and colour of the critical line—red in pure butter, otherwise blue—it can at once be determined, by the aid of the reduction tables supplied with the instrument, whether the butter is natural or artificial. With a little practice, twenty to thirty examinations can be made in the hour.

The relation between the scale index and refractive index is given in the following table :—

Scale Division.	Refractive Index.
0	1·4220
10	1·4300
20	1·4377
30	1·4452
40	1·4524
50	1·4593
60	1·4659
70	1·4723
80	1·4783
90	1·4840
100	1·4895

Genuine butters have been found to vary from 37.5° to 44° , the average being 40.7 at 40° .

At a temperature of 40° C. margarine has an average figure of 42° , coco-nut oil of 34° , palm-nut oil 36.5° , and cotton-seed oil of 58° .

Wollny obtained the following values for natural butter, margarine and mixed butter, expressed in degrees of the scale at 25° C. :—

Natural butter	49.5-54.0 degrees.
Margarine	58.5-66.0 „
Mixed butter	54.0-64.8 „

Butter with a refractometer index of 54.0 at 25° C. is always an object of suspicion, and should be subjected to further examination.

It is simpler, however, to use the ordinary Zeiss refractometer which is exactly like the one already described, only graduated in refractive indices. With this instrument the following figures may be taken as usual :—

	Refractive Indices at 40° C.
Butter fat	1.4506-1.4551
Margarine	1.4473-1.4494
Coco-nut oil	1.4473-1.4497
Palm-nut oil	1.4499
Beef fat	1.4551-1.4588
Cotton-seed oil	1.4646

The complete analysis of butter is performed as follows :—

1. *Estimation of Water.*—5 to 10 grms. of butter are weighed out into a covered beaker and melted in the air oven. When the fat is clear it is filtered (still in the oven) through a tared filter into a tared flask, care being taken to pour through the filter as much of the fat and as little of the underlying water as possible. The aqueous residue is then evaporated in the beaker at 100° C., and the whole of the remaining substance is placed in the desiccator and weighed, dried, cooled and re-weighed until the weight is constant or

varies by not more than 1 mgrm. The loss in weight sustained by the fat—the weight of the beaker being known at the outset—gives the amount of water therein.

The average amount of water in butter ranges from 13 to 15 per cent.; butters with a water content as low as 9 have been met with, while the maximum amount has been 20 per cent. Salt butters, especially those mixed with brine, generally contain 18 per cent. of water. Of late, butters blended with milk have been sold, these containing as much as 22 to 23 per cent. of water. The legal standard of water in butter is however 16 per cent.

2. *Estimation of Fat.*—The residue in the beaker is loosened as completely as possible from the bottom by means of a glass rod and washed with water-free ether on to the filter, which has been placed in a funnel in the mouth of the flask holding the bulk of the fat, the beaker and rod being rinsed and the filter and contents washed with ether until a test drop on a watch glass shows that all the fat is removed. The ether is then distilled off and the fat dried in the air oven at 100° C., cooled in a desiccator and weighed when cold, the operations being continued until the weight is constant. The amount of fat ranges from 82.5 to 86.5 per cent.

3. *Determination of Ash.*—The residue collected on the filter and the filter itself are carbonised in a platinum crucible at a moderate heat and, after extracting several times with distilled water and filtering, calcined thoroughly. The filtrate is then poured by degrees into the ash in the cooled crucible and the whole dried in the water bath, incinerated with the used filter in the (covered) crucible, cooled and weighed, the weight of the two filters being deducted from the result. In the case of salted butter, the total amount of the mineral salts less the amount of ash in genuine butter is approximately the amount of salt present. As, however, butter generally contains no more than 0.10 per cent. of ash, the percentage of

chlorine can only be very small, and the amount of salt used can therefore be very approximately determined from the chlorine in the ash. The presence of preservatives like borax or boracic acid will increase the amount of ash.

To determine the amount of salt the residue or ash of the butter is treated with hot water and the solution filtered. This solution is now titrated with standard silver nitrate using potassium chromate as the indicator. The silver nitrate should be such that 1 c.c. = 0.005 grm. of sodium chloride, standardised against a solution of pure sodium chloride.

4. *Estimation of Protein.*—Eighty to 100 grms. of butter are weighed out and the fat separated completely—after evaporating the water—from the other constituents in the manner already described in the estimation of moisture and fat, the residue, carefully collected on the filter, being used for the determination of nitrogen by the Kjeldahl process; the result multiplied by 6.25 gives the percentage of protein. Bearing in mind, however, that it is doubtful, especially in the case of old butter and that from sour cream, whether all the nitrogenous bodies belong to the albuminoid group, a little uncertainty probably attaches to the figures given for protein.

5. *Estimation of the Soluble Non-Nitrogenous Matter* (milk-sugar, lactic acid, etc.).—The percentage content of water, fat, ash constituents and protein having been precisely determined by two satisfactory and concordant analyses, then the figures thus obtained are added together and the total deducted from 100, the remainder being regarded as expressing the percentage of non-nitrogenous organic matter. According to observations made in the Kaaden Laboratory, attempts to estimate this group directly do not generally attain the desired object, since in the washing of the fat-free residue more or less of the nitrogenous matter passes into solution. The figures ascertained for non-nitrogenous organic matter by difference are also affected by the uncertainty attendant on those for protein.

Examination of the Butter Fat.—For the following tests, the pure dry filtered fat is employed.

6. *Reichert-Meissl Value.*—This test is described on page 298. Treated in the manner described and working on 5 grms. of butter fat, genuine butters require from 25 to 27 c.c. of decinormal caustic soda. Any samples requiring less than 25 c.c. should be regarded with suspicion, although cases are not unknown where genuine butters have used as little as 20 c.c. In such cases other evidence of the genuineness of the butter must be sought for. On the other hand butters requiring as much as 33 c.c. of the alkali have been recorded. Most other fats give Reichert-Meissl values of from 0.5 to 0.7 c.c. but coco-nut fat and palm-nut fats are exceptions, the former requiring 6.7 to 8.4 c.c. and the latter 5 to 6 c.c.

It may be pointed out, however, that it is possible to scientifically adulterate butter by taking one which is known to have a high Reichert-Meissl figure and adding to it just sufficient margarine to bring it to the lower limit but no more.

Polenska Value.—By means of the Hehner and Reichert-Meissl values it is possible to determine in many cases whether a sample of butter is genuine, but since the introduction of coco-nut oil and palm-nut oil into margarine manufacture the addition of a small quantity of the latter has become increasingly difficult to detect, as may be seen from the following figures:—

	Hehner value.	Reichert-Meissl value.
Butter fat	85.90	26.30
Coco-nut fat	88.6-90.5	6.7-8.4
Palm-nut fat	91	5.7-8
Oleomargarine	95.96	0.5-0.7

It has been noticed, however, that when the total fatty acids are distilled along with water a small quantity of insoluble acid separates out on the surface of the liquid.

In the estimation of the Reichert-Meissl value this is filtered off before titration, but it was soon found that the

quantity of this insoluble volatile acid varied with the fatty acids which were distilled, and attempts were made to utilise this factor for determining the nature of the fat under observation, the most successful process up to the present being that devised by Polenska which is described on p. 298.

The Polenska values of butter fat, coco-nut fat and palm-nut fat are given below.

	No of c.c. of $\frac{1}{10}$ N. KHO req. for 5 grams of the fat.
Butter fat	2.3-3.3
Coco-nut fat	15-20
Palm-nut fat	10-12
Most other oils and fats	not more than 0.5

As emphasised in the description of the process (p. 298) it is absolutely necessary to follow the same procedure in all cases, otherwise the results will be quite conflicting.

Rate of Distillation of the Volatile Acids.—E. Duclaux in an elaborate investigation on the volatile acids of wine showed that the rate of distillation of each volatile acid was characteristic. That is to say, no matter what the concentration of the acid may be, the amount found in the distillate at any given moment was always a constant quantity of the total acid present, and that the rate of distillation remained the same even in mixtures, so that, provided the two acids were known, the percentage composition of the mixture could thus be determined. Of course the rate of distillation will vary with the barometric pressure, but for the sake of simplicity the experiments are performed at the ordinary pressure. Duclaux took 110 c.c. of the liquids and distilled 100 c.c. in fractions of 10 c.c., titrated each fraction separately and then calculated the proportion of acid in each fraction (A) on the total acid distilled and (B) on the total acid in the distillation flask. The following figures will illustrate this :—

	Formic Acid.		Acetic Acid.		Butyric Acid.		Caproic Acid.	Caprylic Acid.
	A.	B.	A.	B.	A.	B.	B.	B.
1.	5'5	3'5	7'2	5'5	16'8	16'1	33'5	55'5
2.	11'9	7'6	14'7	11'2	31'9	31'1	56'0	78'0
3.	18'5	11'8	22'7	16'9	45'4	44'3	75'5	91'0
4.	25'7	16'3	31'2	23'7	57'7	56'1	86'0	93'0
5.	34'0	21'6	40'2	30'6	67'9	66'2	92'5	95'0
6.	43'1	27'3	49'9	32'9	77'2	75'3	96'5	96'8
7.	53'1	33'7	60'2	45'8	85'0	82'9	97'5	97'8
8.	65'2	41'4	71'9	57'7	91'4	89'2	98'4	99'0
9.	79'8	50'7	85'3	64'8	96'4	94'0	99'3	99'5
10.	100'0	63'5	100'0	75'9	100'0	97'5	100'0	100'0

It will be seen that the rate of distillation increases with the molecular weight.

The accuracy of the process may be gauged by comparing the figures obtained by the reviser and another, given below, with those of Duclaux. *Acetic acid.* 1. contained 3'5 c.c. strong acid in 500 c.c.; 2. contained 2'5 c.c. in 500 c.c.; 3. 2'5 c.c. glacial acid in 500 c.c.; the others contained the specified percentages. As 110 c.c. is a small amount to distil, in numbers 1, 2 and 3, 275 c.c. were taken and 25 c.c. collected at a time, in the other cases 165 c.c. were taken and 15 c.c. collected in each fraction. Only the A figures are given:—

Acetic Acid.	1.	2.	3.	0'1 Per Cent.	1 Per Cent.	2 Per Cent.	5 Per Cent.	10 Per Cent.
1.	7'9	7'8	7'5	6'15	7'62	7'56	7'91	7'90
2.	15'6	15'8	15'3	13'46	15'77	15'64	16'11	16'26
3.	23'8	24'0	23'2	21'83	24'29	24'02	24'62	24'87
4.	32'4	32'7	31'2	30'80	33'02	32'72	33'41	33'73
5.	41'6	42'0	40'9	40'03	42'04	41'96	42'71	43'00
6.	51'3	51'5	50'7	50'31	51'54	51'50	52'31	52'70
7.	61'0	61'7	61'0	60'79	61'74	61'74	62'55	62'87
8.	72'8	72'8	72'2	72'39	72'83	72'84	73'46	73'83
9.	85'3	85'3	84'7	85'04	85'16	85'27	85'72	85'91
10.	100'0	100'0	100'0	100'00	100'00	100'00	100'00	100'00

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Formic Acid.	1.	2.	0.1 Per Cent.	1.0 Per Cent.	10 Per Cent.
1.	6.5	5.7	5.73	6.13	5.67
2.	12.3	11.3	11.92	12.55	11.87
3.	18.8	18.6	18.74	19.22	18.56
4.	25.7	25.7	26.11	26.53	25.85
5.	33.5	33.5	34.07	34.62	33.79
6.	42.1	42.5	43.17	43.43	42.47
7.	52.2	52.6	53.73	53.44	51.88
8.	63.9	64.7	65.97	64.97	62.77
9.	78.7	79.4	81.17	79.45	77.01
10.	100.0	100.0	100.00	100.00	100.00

This process should form a very excellent one for butter and margarine if properly worked out, and some attention has recently been given to the subject. Some tentative experiments by the reviser in this connection may not be devoid of interest. 50 grams of the fat were saponified with 15 grams of KHO and alcohol. After evaporation of the alcohol, 8 grams of sulphuric acid in water were added, the insoluble fatty acids separated and washed and the filtrate and washings made up to 500 c.c. 275 c.c. were distilled and the distillate collected in fractions of 25 c.c., each being titrated with $\frac{N}{10}$ NaHO and the acid in each fraction calculated to a percentage of the whole distilled, two experiments in each case:—

	Butter.		Neutral Coco-nut Fat.		Palm-nut Fat.	
	1.	2.	1.	2.	1.	2.
1.	25.9	22.4	42.4	42.8	28.6	31.2
2.	43.4	40.8	67.4	67.2	48.2	49.9
3.	57.7	56.1	81.0	80.2	60.8	63.8
4.	69.6	68.5	88.0	87.2	69.2	71.5
5.	79.0	78.3	92.7	91.6	75.5	77.0
6.	86.4	86.0	95.2	94.4	81.1	81.2
7.	92.0	92.0	96.5	96.0	86.0	85.4
8.	96.0	96.0	98.1	97.9	90.9	89.6
9.	98.5	98.5	100.0	100.0	95.1	93.8
10.	100.0	100.0	—	—	100.0	100.0

The distillations are difficult towards the end owing to the bumping which occurs; this explains why those of coco-nut fat were stopped after the ninth fraction had passed over.¹

It may also be of interest to note that Elsdon found by Haller's method (fractional distillation of the methyl esters in palm-kernel fat: caproic acid 2, caprylic 5, capric 6, lauric 55, myristic 12, palmitic 9, stearic 7, and oleic 4 per cent.; and in coco-nut fat: caproic 2, caprylic 9, capric 10, lauric 45, myristic 20, palmitic 7, stearic 5, and oleic 2 per cent. These figures may be compared with those of butter fat given on p. 103.

7. *Hehner Value*.—This test is described on page 298. Butter contains from 85 to 90 per cent. of insoluble fatty acids, while tallow and most other fats contain 95 to 96 per cent.

Coco-nut fat yields 88.6 to 90.5 per cent. of insoluble fatty acids, and palm-nut oil about 91 per cent. It must be remembered, however, in this connection that any unsaponifiable matter contained in the original fat is separated and estimated along with the insoluble acids.

8. *Kötstorfer's Saponification Test*.—This is described on page 297. Genuine butters require for 1 gram of fat 220 to 233 milligrams of caustic potash (KHO) for complete saponification. Most other oils and fats absorb from 190 to 198 per cent., with the exception of coco-nut and palm-nut oils, which require 242 to 260 per cent.

Preservatives in Butter.—For the purpose of preventing butter becoming rancid too quickly preservative agents have been added. The most common are borax and boracic acid; sulphites and nitrates have also been used, also salicylic acid, salicylates, and benzoic acid. Formalin (formic aldehyde) has been used to a small extent. Borax and boric acid may be detected by melting the butter and allowing the saline and

For further particulars on Duclaux's method, see Winter, Blyth, *Foods, Composition and Analysis*, 1882.

aqueous matters to settle out. A piece of turmeric paper dipped in the aqueous liquid will turn red if boracic acid be present, while borax is shown by adding a little hydrochloric acid before dipping in the turmeric paper, which will then turn red. The amount of boron preservative is estimated by a modification of Thomson's process, as revised by Richmond and Harrison. A weighed quantity of butter (about 25 grams) is placed in a stoppered cylinder, sufficient water is added to make the total water 25 c.c. and then 10 to 15 c.c. of chloroform. The whole is shaken and then allowed to settle. A measured portion of the aqueous liquid is run off into a platinum dish, rendered alkaline with a little soda, then evaporated and ignited. The residue is dissolved in 25 c.c. of hot water, rendered faintly acid with dilute sulphuric acid using methyl orange as indicator, it is then boiled for a few seconds to expel carbonic acid, neutralised to a faint orange with a weak solution of caustic soda, then 25 c.c. of glycerine and phenol-phthalein are added and the solution titrated to a pink tint with $\frac{1}{10}$ N. NaHO. 1 c.c. of this solution = 0.0062 gram of H_2BO_3 . Sulphites can be detected by sulphur dioxide being evolved on the addition of acids. Nitrates by the application of the diphenylamine test. Formalin is detected by Hehner's test as modified by Richmond and Bosely. The butter is melted at a low heat and the lower aqueous layer run off, it is mixed with a few drops of milk and a drop of a weak solution of ferric chloride, and is then carefully poured upon the surface of some strong sulphuric acid contained in a test tube, when if formalin be present a violet-blue ring will be formed at the junction of the two liquids.

Salicylic acid is readily detected by shaking the butter with a little alcohol and a few drops of a weak solution of ferric chloride, the liquid which separates being of a violet tint if salicylic acid is present.

Benzoic acid and benzoates may be extracted by the method of Fischer and Gruenest: 150 grms. of the butter is melted and shaken with a 1 per cent. solution of bicarbonate of soda; the aqueous solution is run off, neutralized by the addition of dilute sulphuric acid, and a few drops each of solutions of copper sulphate and caustic soda are added, the liquid is then slightly acidified and filtered. The filtrate is shaken up with ether in a separator and the aqueous liquid run away, the ether being washed two or three times with water. The ethereal solution is dried over calcium chloride and distilled at a low temperature, leaving the benzoic acid in a crystalline condition. The benzoic acid can be recognised by dissolving in ammonia, boiling off the excess of ammonia, and then testing with a drop of Fe_2Cl_6 solution, which, after boiling, yields a red precipitate. In the absence of salicylic acid it may also be detected by oxidising it to salicylic acid by adding hydrogen peroxide, ferrous sulphate, and ferric chloride, when a violet colour will be produced (Jonescu and Fleury).

Fluorides are also used to some extent as preservatives for butter and margarine; they are detected by melting 50 or 100 grms. of the material, separating the water, evaporating this to dryness. After addition of a little caustic soda, the residue is then brought into a platinum crucible, treated with strong sulphuric acid and covered with a watch glass protected with a coating of paraffin wax which has a design marked through the wax so as to leave some of the glass uncoated. In a few hours, if fluorides are present, the glass will be distinctly etched.

Boric acid and borax are the chief preservatives used, and according to a Government Laboratory Report, 53.5 per cent. of the Canadian butters, 93.8 per cent. of the French, 93.6 per cent. of the Belgian, 83.3 per cent. of the Australian, 84.4 per cent. of the South American, and 79.5 per cent. of the New Zealand butters examined in these laboratories con-

tained boron preservatives; also of 364 samples of butter (both home and foreign) examined, 208 were found to contain boron preservatives, and of 133 samples of margarine 99 were similarly preserved. The amounts varied from 18 to 65 grains per lb. in butter, and 7 to 73 grains per lb. in margarine.¹

Microscopic Examination of Butter.—If a small portion of butter be placed on a microscope slide and a cover glass be placed over it and pressed down so that it forms a slightly wedge-shaped film, being rather thicker at one side than the other, on examination of the sample under a $\frac{1}{6}$ th in. objective, using the polariscope and a selenite plate, it will be noted, with a selenite plate giving alternately a green and a red field, that with pure butter the field will be uniformly green and red as the polariscope is revolved, but with an adulterated sample or with margarine the field will be mottled green, blue, and red in patches, especially in the thicker part of the sample. This is due to the fact that butter, not having been melted, is a homogeneous fat, whereas margarine, which has been melted, contains crystals, of different composition to the bulk, which affect the polarised light. Of course, if butter be melted and allowed to cool it will also show the same play of colours, therefore in such a case the test would be of no value. Rancid butters also sometimes show a slight mottled appearance.

Ghi or Ghee.—In India the butter from cows' or buffalos' milk is heated for about 12 hours over a fire until all the water has evaporated and the casein and salt have settled to the bottom. After the fat has cooled somewhat it is poured by decantation into an earthenware vessel in which it is stored for some time before being used. Ghi is regarded by the natives as a very valuable product and is esteemed as a great

¹ Report of the Departmental Committee on the Use of Preservatives and Colouring Matters in Food, Cd. 883, 1909.

luxury. It is often kept for long periods; in fact, it is affirmed that ghi 100 years old or even more is not at all uncommon. In the year 1672 Feyer records that he was shown some tanks of butter 400 years old, and that it was black and stinking.¹ There is a considerable amount of "ghi" produced in India and a flourishing trade exists between the different states and also with contiguous countries, but none of it comes to Europe. The price in India is Rs. 1 8 to Rs. 1 14.

Three samples of ghi, two from buffalos' milk and one from cows' milk, were forwarded to the Imperial Institute by the Director-General of Commercial Intelligence in 1908, and their examination yielded the following results:—

	Ghi from Pure Cows' Milk.	Ghi from Buffalos' Milk.	
		Pure.	Ordinary.
Moisture	2.0	0.4	0.5
Curd	0.35	0.0	0.0
Constants of the fat:—			
Specific gravity at $\frac{100^{\circ}}{15^{\circ}}$ C.	0.864	0.866	0.864
Acid number	7.24	5.46	11.1
Saponification number	222	226	224
Iodine number	34.8	34.6	35.0
Solidifying point	30.2° C.	29.4° C.	26.1° C.
Reichert-Meissl number	24	31	29

ARTIFICIAL BUTTER, OLEOMARGARINE, ETC. (KUNST-BUTTER, MARGARINBUTTER, OLEOMARGARINE, SPARBUTTER).

By the above and similar designations is understood an artificial product obtained from constituents of beef tallow, prepared by special processes and worked up with milk and vegetable oils to a substance greatly resembling, or even identical with, natural butter in appearance, consistency and flavour.

¹ Sir George Watt, *The Commercial Products of India*, 1908, p. 478.

The manufacture of artificial butter is a fairly simple process, but nevertheless entails the exercise of intelligence and care in order to fulfil all the requirements imposed. A few of the methods in use are given below, the reader being referred for fuller information to Lang's work on *Kunst-butter* (3rd edition, 1895).

Margarine Manufacture in France.—Margarine was originally made in France by Mège Mouriès in 1870 as the result of a competition for a prize offered by the French Government in 1869 for the production of a suitable substitute for butter, which at the time of the Franco-Prussian war had become very scarce. The method of making this artificial butter devised by Mège Mouriès was to heat fresh beef fat, especially the kidney fat, with a solution of carbonate of soda together with a small quantity of the stomach of the pig or sheep. The pepsin contained in the latter digested the membranous matter, liberating the fat, which was skimmed off, warmed with a solution of common salt to preserve it. The fat was then cooled to 22° C. and pressed between warm plates when a soft fat exuded, to which the name "oleo-margarine" was applied. Subsequently the process was improved by churning the soft fat with 10 per cent. of milk and 0.4 per cent. of macerated cow's udder until a perfect emulsion was formed. This produce contained about 12½ per cent. of water and had a melting-point of 17° to 20° C.

Villon describes the process as follows:—

The first operation consists in the separation of the so-called *premier jus* from the suet by melting at 38° C. The solid beef fat is formed into pats, about 8 in. long by 7 in. wide and ½ in. thick, wrapped in linen cloths, and weighing some 2½ lb. (1 kilo) each. Two hundred of these pats are placed in a hydraulic press by placing on the lower plate (covered with a canvas cloth warmed to about 50° C.) a layer

of 5 pats, these being covered with another cloth also warmed to 50° C. and surmounted in turn by a second set of 5 pats, and so on until the whole 200 are in position. The operations must be performed quickly, so that the cloths have not time to become cold. The separation of the oleomargarine begins without pressing, owing to the pressure of the cloths and to the warmth to which they are exposed, but as soon as the flow is observed to flag, the press is set to work, very slowly at first, the pressure being afterwards raised to 150 kilos (330 lb.) and even to 175 kilos (385 lb.).

The pressed beef stearin (*suif pressé*) remains behind in the cloths and is used for making stearin and tallow candles. The oleomargarine or oleo oil thus obtained is a yellow crystalline mass resembling ordinary butter, it is mixed with milk and cotton-seed oil or ground-nut (*arachis*) oil for churning. The proportions taken vary according to the quality in view, but the following have been used:—

	I.	II.
Oleomargarine . . .	800 kilos	500 kilos (of 2·2 lb.).
Milk	500 litres	500 litres (of 0·22 gall.).
Cotton-seed oil . . .	100 kilos	50 kilos.

The quantity of oil is adjusted according to the time of the year, 30-40 per cent. being used in winter, whilst in summer the oil is occasionally omitted altogether. Sesame oil is sometimes used, the object of the oil, of whatever kind, being to impart to the oleomargarine the suppleness of butter.

The oleomargarine is melted at 45° C., and the milk and oil are added at the same temperature, all being run into the churns through pipes from storage tanks several yards high, situated above the (wooden) churns. The materials are then thoroughly incorporated by means of dashers, the operation lasting about two hours, the dashers being stopped towards the end in order that the quality of the product may be determined. The mixture is then allowed to run out of the

churn through an inclined wooden trough into a large vat, a jet of cold water impinging on the mixture, as it falls from the trough, in order to granulate the margarine. In the vat the product floats in cold water, and is removed by gauze strainers or sieves and left to drain for about two hours in perforated vessels, whence it is transferred to revolving tables like those used for butter working, only larger. On these it

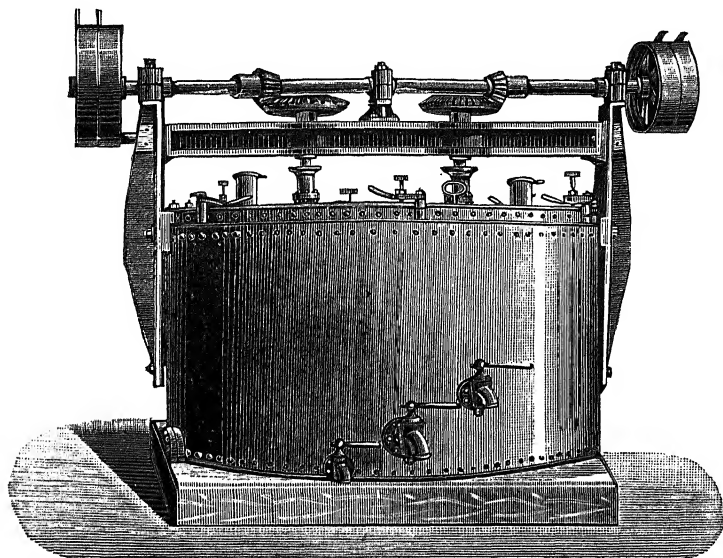


FIG. 52.—Butter and churning machine (W. Rivoir, Offenbach-am-Main).

is kneaded to express the milk, and the margarine thereby acquires the homogeneity of butter. The appearance of the product is improved by passing it in small portions through horizontal rotating fluted-roller machines, after which it is made up into 1 lb. (or $\frac{1}{2}$ kilo) pats. This method is the one most frequently employed, but the churned mixture can also be cooled by ice water from the ice-making machine, though this plan is not advantageous, since, during the slight fermentation allowed to ensue before the mass is drained, the flavour,

sui generis, of a good margarine, is, in this case, not attained.

To obtain really good margarine the product must be allowed to set in small lumps, enclosing small quantities of milk, and on this account the water used must not be too cold. A good butter flavour is developed by inoculating the margarine in the draining stage with a small quantity of a

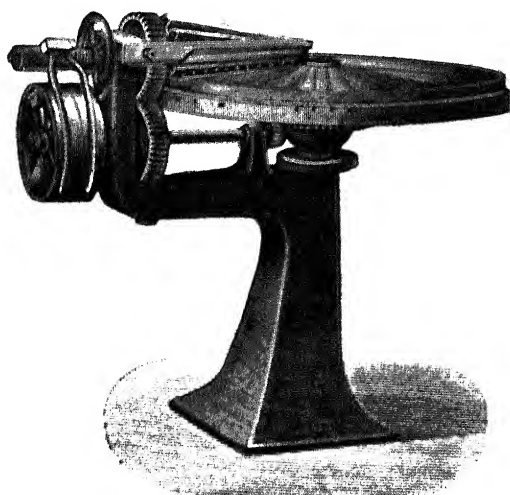


FIG. 54. Margarine worker (W. Rivoir).

ferment obtained from artificial fermentation-cultures from good butter.

Henri Grasso of Herzogenbusch (Holland) designs and supplies margarine factories for a diurnal production of 1-3 tons of artificial butter. The entire plant consists of;—

1. Steam-heated churns of 330-550 galls. (1500-2500 litres) capacity;
2. Roller workers with patent "Salamander" rollers, filling sack, flap and truck;
3. Margarine oil attemperator and mixers;

4. Margarine melting tanks with agitators ;
5. Butter driers or mixing machines ;
6. Milk acidifiers, holding 50-120 galls., fitted with heating and cooling arrangements ;
7. Milk separators ;
8. Pasteurising apparatus, several coolers for cream, skim milk and pasteurised milk ;
9. Milk heater holding 70-115 galls. ;
10. Churn for natural butter or vegetable margarine ;
11. Margarine oil pumps, worked by hand or power ;
12. Granulating sprays, with taps ;
13. Kneading tables ;
14. Mixing or blending machines ;
15. Cooling drums ;

The following particulars relating to the manufacture of margarine are taken from a paper by W. Clayton in the *Journal of the Society of Chemical Industry*, 1917, p. 1205 : The milk is pasteurised by heating for a few minutes at 82° C., which destroys most of the bacteria present ; it is then run over a vertical cooler to the souring tanks, where it is inoculated with a specially prepared pure culture of lactic bacteria, this being known as the "starter". The temperature of the tank is kept at the most suitable point for the development of the bacteria which cause rapid curdling owing to the formation of lactic acid. The fats are melted in large oval jacketed, steam-heated vessels and run into the churn. The churns are of two types, the older form being double-jacketed, provided with two sets of stirrers provided with paddles which agitate the contents. The newer continuous electric churns are very efficient. The oil and milk are now added and when emulsification is completed at a temperature of 25°-35° C. the mixture is run from a valve at the bottom into a sloping shoot where it is met with a spray of ice-cold water which impinges upon it with such force as not only to solidify it but to break

it up into a number of minute fragments or "crystals". In an alternative method the margarine from the churns is run on to revolving cooling drums from which it is automatically removed by means of a knife or "doctor". The "crystals" or paste is kept for a time in a "maturing" room in which the temperature is kept at a definite point favourable to the growth of the organisms innoculated into the milk. The margarine is then kneaded to bring it into a homogeneous mass and to expel the excess of water, the legal limit of the latter being 16 per cent. The margarine is then blended and mixed with

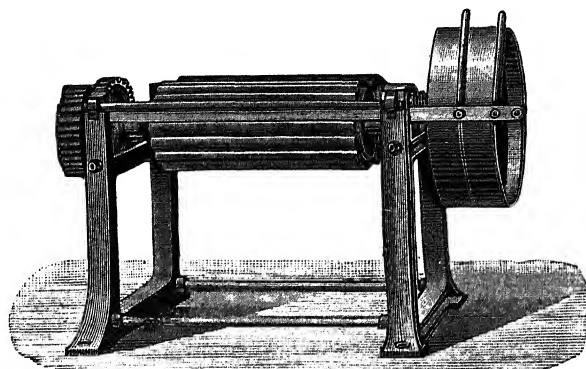


FIG. 54.—Butter mill with fluted rollers.

salt, colouring matter, and preservative in a blending or kneading machine and then placed in kegs or moulded into pats. Preservatives, usually boric acid, are almost universally employed, the amount not exceeding 0.5 per cent., with the object of preventing further bacterial action, which would cause sourness or rancidity. The constituents of margarine are various. At first a blend of oleo oil and cotton-seed oil was employed, but the cheaper brands of margarine are now made from neutral coco-nut fat or palm-kernel oil without any animal fat. In the United States, in addition to cotton-seed oil, arachis or earth-nut, soya bean, sesame, and more recently kapok, maize,

and wheat oils have been employed. In the better class of margarines a small proportion of butter is added, but in this country, in order to comply with the law, the amount must not exceed 10 per cent.

Hydrogenised oils are now employed in the manufacture of margarine, these being made from any oil—including fish oils—of any degree of consistency, from that of lard to that of tallow, according to one of the methods outlined on pp. 283-291. These hardened oils are white and usually quite free from odour and are somewhat difficult to detect; they usually, however, contain a trace of nickel, which can be detected as described on p. 293. The amount of nickel present in hardened oils is now much smaller than it was originally, and the question is whether this amount is likely to prove injurious if taken internally over a long period. Much controversy has arisen over this problem, it having been affirmed and denied that such small doses may be taken with impunity. It is said that 98 per cent. of the nickel thus ingested is quickly eliminated. The hydrogenated oils are digested in the same way as ordinary fats, and from this point of view no objection could be taken to their use.

Other materials are added to margarine in order to cause frothing and browning and thus simulate butter; among these substances may be mentioned butter, dried milk powder, casein, egg yolk, lecithin, yeast, malt extract, lactic acid, and various proprietorial preparations.

The German and Austrian Margarine Acts specify that manufacturers must use 10 per cent. of sesame oil in admixture with the other oils in order that the product may be readily distinguished from butter. The Belgian Act also stipulates 5 per cent. of sesame oil and 1·2 per cent. of potato starch, calculated on the weight of the fats, with the same object.

Kaiser Butter.—According to a communication issued by H. & M. Pataky, patent agents of Berlin, it has been

established on the basis of scientific experiments that levulose and substances containing the same, when incorporated with edible fats by a special process, yield a preparation that, unlike other fats, needs no admixture with bile to form an emulsion in the alimentary canal. All edible fats liquefy, it is true, at the temperature of the body, but their emulsifying capacity is dependent on their intermixture with the biliary fluid in the intestines, and since the amount of this fluid is in most persons comparatively small, the ordinary edible fats are only to a small extent actually digested, *i.e.*, absorbed by the intestines. Of all the alimantal fats hitherto known, good

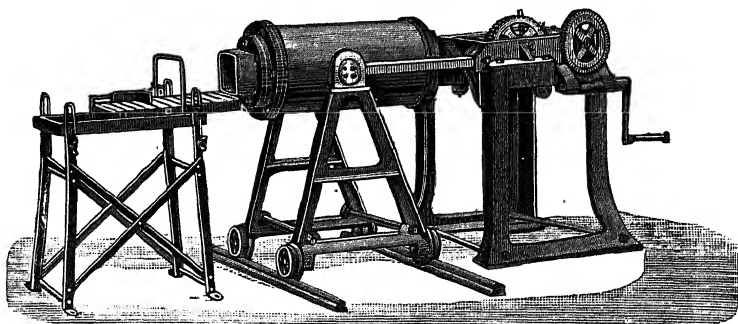


FIG. 55.—Moulding machine for margarine (W. Rivoir).

fresh butter is the easiest of digestion, owing to its superior capacity—in comparison with the others—of emulsifying with relatively low proportions of bile. If, now, edible fats be incorporated with levulose, they liquefy in the human stomach and form emulsions with water at temperatures even below 37° C., so that their digestion is thus ensured. The process is carried out in such a manner that the fats, warmed to about 70° C., are mixed with an addition of a solution of levulose at the same temperature, the mixture being rendered thoroughly intimate by continual stirring until cold, so that it emulsifies when shaken or otherwise agitated with water at temperatures from about 14° C. upwards. Butter mixed with levulose (or

substances containing the same) by this process is allowed to be sold as butter in Germany, and a patent has been granted for the "Kaiser butter" prepared in this manner. In addition to the properties already enumerated, "Kaiser butter" exhibits the advantages of superior flavour and keeping qualities. Consequently, the consumption of butter would be increased, and that of the butter substitutes at present vended decreased, by the introduction of this preparation, the price of which is even lower than that of ordinary butter.

Preparation of Alimentary Fats, according to Jahr and Münzberg.—According to the above-named inventors, easily digestible alimental fats may be prepared by intimately mixing fat, warmed to 70° C., with a solution of levulose at the same temperature, the liquids being stirred without interruption until cold. The mass thus obtained can be emulsified by shaking or otherwise agitating it with water at temperatures ranging from about 14° C. upwards.

Filbert's Process.—J. H. Filbert's process for preparing alimental fats consists in the admixture of beef fat and cotton-seed oil, the former being gradually liquefied at the lowest possible temperature and the oil added, whereupon the mixture is vigorously stirred and beaten in order to reduce the size of the oil globules still further and bring about their intimate incorporation. Simultaneously, air is blown through the mixture, so that the finished product is light in colour and the particles of cotton-seed oil and minute air bubbles are held in suspension in the solid fat.

Winter's Method.—A. M. Winter took out an American patent for the following mixtures as alimental fats (! ! !):—

1. Fatty substances and refined mineral oil mixed in such proportions as will produce a stiff mass.
2. Refined mineral oil and animal or vegetable fats in proportions calculated to form a stiff mass.

3. Refined mineral oil, oleostearin, tallow and vegetable oil (cotton-seed oil).

4. Sixty per cent. of refined mineral oil and 40 per cent. of fatty substances.

5. Sixty per cent. of refined mineral oil, 30 per cent. of animal and 10 per cent. of vegetable fat.

6. Sixty per cent. of refined mineral oil, 15 per cent. of oleostearin, 15 per cent. of tallow and 10 per cent. of cotton-seed oil.

Analysis.—The analysis of margarine is carried out exactly as for butter, see p. 108.

In the analyses of margarines consisting of vegetable oils (coco-nut, palm-nut) admixed with butter it is often somewhat difficult to determine by means of the Reichert-Meissl or Hehner values whether the legal limit of 10 per cent. of butter has been overstepped. In such cases the Polenska number, that is the amount of alkali required to neutralize the insoluble volatile acids, will be found of great service. This is, however, an empirical method, and in order to obtain concordant results it is necessary to proceed always according to a uniform plan; even the size of the distilling flask and the other parts of the apparatus have had to be standardised. For a full account of this method see Lewkowitsch, *Chemical Technology and Analysis of Oils, Fats, and Waxes*, 1915, Vol. III, pp. 35 *et seq.*

LARD (SCHWEINEFETT, SCHMALZ).

Raw Material.—The fatty portions of the hog, and especially those lying beneath the skin and between the intestines, the leaf. The quality of the fat varies with the part of the body from whence it is derived; that from the exterior fatty integument of the hog, surrounding the entire carcase of the animal immediately below the skin (especially on the back and sides), is more solid and curdy, though, at the same time,

more easily melted out than that from the interior of the body along the ribs, the intestines and kidneys, which is generally known under the names of leaf, loin, kidney fat (Schmeer, Liesen, Lendenfett, Nierenfett, Filz). The proper preparation and assortment of the raw material for preparing the lard is very important. January and February are the best months, the fat being then more consistent than during the warmer seasons of the year. Apparently the colder season, as a result of increased respiration of the denser air, has an influence in rendering the deposited fat poorer in hydrogen and richer in carbon, the hydrogen, by reason of its greater affinity for oxygen, being the first to enter into combination with the latter during respiration at low temperatures. Winter fat is accordingly richer in stearin, whilst summer fat contains more olein. The method of feeding and the health of the animal also exert a not unimportant influence on the constitution of hog fat, so that soft and greasy fat is also met with in winter. Pigs fed exclusively on acorns always yield a somewhat yellowish fat. Lard in the greatest quantity and of the best quality, both as regards consistency and appearance, is supplied by the intestinal fat of young pigs, this fat consisting mainly of cell capsules enclosing the pure fat and requiring to be broken before the melting process.

Preparation.—Lard is prepared in the same manner as tallow, by carefully melting the raw material and separating it from the cellular tissue and other residual fleshy matter. The fatty substance intended for melting is freed as much as possible from all adherent pieces of skin, sinews and flesh, and then cut into small cubes with knives or in the cutting machine, the material so prepared being repeatedly washed with water until the washings run away clear and colourless, after which it is transferred to the melting pan. This is generally of well-tinned copper, and is heated by direct fire, though steam-jacketed enamelled iron or large stoneware

pans may be employed. The melting fat is constantly stirred with large wooden paddles, the work being continued until the liquid has become perfectly clear and all the water is evaporated. The clear fat is then poured through straining cloths, stretched on frames, into the storage or transport vessels, and after being slightly cooled is stirred until it has become white and opaque, although still thickly fluid. If the fat were allowed to simply set by itself, the contraction, occurring in congelation would give rise to fissures in the mass, which would admit air to the interior and thus unfavourably influence the keeping quality. When, on the other hand, it is stirred until completely set, then so much air is stirred in that all cannot escape, and rancidity is still further facilitated. The stirring must therefore be discontinued at the proper time, whilst the fat is still soft enough to run together without leaving any interstitial spaces. When this particular method of cooling is not adopted a quantity of oily fat congregates on the surface and, being readily liable to alteration by rancidity, exerts an unfavourable influence on the consistency of the subjacent mass.

The residual matters (fragments of tissue, greaves) left behind in the straining cloths and in the pan are heated again in the pan to a somewhat higher temperature and then pressed in a so-called greaves-press, leaving the solid portions behind as greaves. The resulting fat is generally somewhat coloured and possessed of a peculiar roast smell. In the large hog-slaughtering establishments in America the fat is generally melted in apparatus similar to that described on pp. 43 *et seq.*, and heated by steam under pressure, the precautions necessary in the case of tallow melting being, however, dispensed with, owing to the absence of any unpleasant smell.

The greaves are used as an inferior food stuff and fed to cattle, or, when obtained in large quantities, employed for the manufacture of potassium, ferrocyanide.

Chemical and Physical Constants of Lard.—The constants of lard vary very considerably according to the portion of the animal from which it is obtained and also the method of feeding the hogs, but the following numbers may be taken as representative:—

Specific gravity at 15° C.	0·931-0·938
Specific gravity at 40° C.	0·8985
Solidifying point	27·1°-29·9°
Melting-point	36°-46°
Free acid	·14-·26
Saponification number	195·2-196·2
Iodine number	49·9-70·4
Refractive index at 40° C.	1·4558-1·4640
Hegner number	95·8
Insoluble Fatty Acids—	
Solidifying point	34°-42°
Melting-point	35°-44°
Iodine number	56-67
Refractive index at 40° C.	1·4545-1·4558.

The characters of American lard and especially of Chinese lards differ very greatly from European lards, which is ascribed to the method of feeding; thus American and Chinese hogs are fed largely upon maize, which renders the fat softer and more fluid, the iodine number being correspondingly higher. Thus European lards have iodine numbers of 52 to 60·5, American lards 59 to 70, and Chinese lards 58 to 85; nevertheless, a lard with iodine value of less than 50 or over 68 should be regarded with suspicion and requiring very careful examination.

Lard consists of olein, palmitin and stearin, and contains, according to Allen and Thomson, 0·23 per cent. of unsaponifiable matters. The olein content—as calculated from the iodine number of 59·0—is 68·4 per cent.; Braconnet states it as 62 per cent. Linolic acid, however, is also present. Twitchell has calculated that a lard having acids with iodine value 62·57 contained:—

Linolic acid	10·06
Oleic acid	49·39
Solid acids (by diff.)	40·55

Lard very quickly becomes rancid on exposure to air, and in that condition will absorb copper or lead from coppered vessels or those coated with lead enamels, and thereby becomes poisonous. To test for these metals the fat is digested with acetic acid, and a part of the acid liquor treated with yellow prussiate of potash (potassium ferrocyanide). A brown coloration indicates copper, whilst lead is revealed by the white precipitate produced by dilute sulphuric acid in a second portion of the acid extract.

Lard is soluble in 27 parts of absolute alcohol or 35 parts of alcohol of sp. gr. 0.9128, also in ether, chloroform, carbon bisulphide, hot benzine and warm petroleum spirit. When fresh, it has a neutral or very faint acid reaction, and yields on saponification and subsequent decomposition of the product 10.5 per cent. of glycerine and 95.6 per cent. of stearic, palmitic and oleic acids. Of this 106.1 per cent., 6.1 per cent. represents water absorbed in the production of glycerine and acids.

The colour of lard is white, granular and of salvy or pasty consistency, with an agreeable, rather sweet, fatty taste. It runs when held between the fingers, and on re-cooling after melting assumes a crystalline structure. It may be separated by pressure at 0° C. into a liquid lard oil (schmalzöl, specköl, huile de graisse), and a solid portion, lard-stearin, the latter product finding employment for stiffening fluid lard.

Adulterations.—One of the most common methods of falsification is the incorporation of an excess of water to increase the weight. The readiest means of detecting this is by carefully melting a sample and leaving it to stand in a moderately warm place, whereupon the water will separate out. With traces of water the lard remains turbid. For increasing the weight of the fat the following additions are used: soda solution, potash solution and soda lye, all of which whiten the fat; also chalk, alum, clay, common salt and starch. Further-

more, alum or lime is added to lards already mixed with water, 1 per cent. of lime or 2-5 per cent. of alum being able to mask the presence of 12 or 15 per cent. of water. Other fats, notably beef stearin, are also added, and in America adulteration with cotton-seed oil was at one time practised on a large scale.

Examination.—The method of detecting water has already been described. To identify chalk, clay or alum 100 grms. of the fat is melted at 69°-70° C. along with the same amount, or half as much again, of water, frequently stirred, and then set aside for a few hours. *Chalk* or *clay*, if present, will subside; *common salt* (sodium chloride) will be revealed by the white precipitate produced in the aqueous solution by acid silver nitrate; *alum*, by the voluminous precipitate with ammonia and the white precipitate with barium chloride; *soda* or *potash solution* or *lyes*, by the precipitates with calcium chloride; and the last-named substances, in particular, by the yellow coloration given with mercuric chloride.

Admixtures of *starch* and *meal* give a blue coloration with iodine solution. Occasionally, beef fat is employed to falsify lard, and can be identified by microscopic examination (Belfield test), since, whereas lard crystallises from an ethereal solution as rhomboidal lamellæ with the ends cut off obliquely, the crystals of beef fat are plume-shaped or curved like an S. An idea of the relative proportions of the two constituents can be obtained from the appearance of the crystals. (Figs. 56 to 61).

According to Schaedler, the manufacture of lard for alimentary purposes, pursued on such a large scale in America, has, unfortunately, led to the shipment to Europe of enormous quantities of lard falsified by the addition of other fats. It is only in quite recent times that this adjunctive adulteration industry has somewhat abated, as a result of the operations of the Food and Drugs Act in this country and stringent

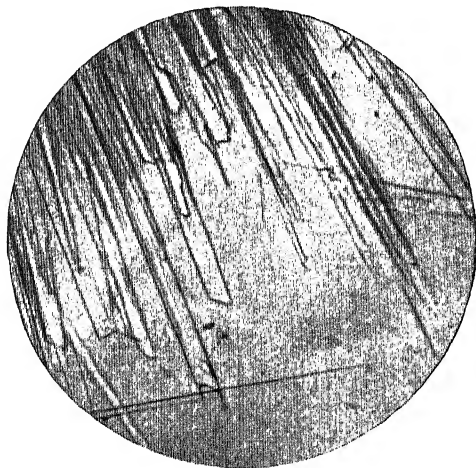


FIG. 56.—Lard stearin, fourth crystallisation $\times 170$ dia.



FIG. 57.—Beef stearin, fourth crystallisation $\times 170$ dia.



FIG. 58.—Lard adulterated with beef stearin $\times 50$ dia.

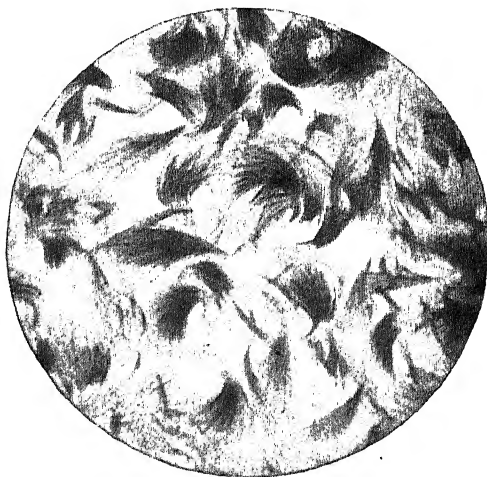


FIG. 59.—Beef fat $\times 50$ dia.



FIG. 60.—Lard crystals $\times 170$ dia.

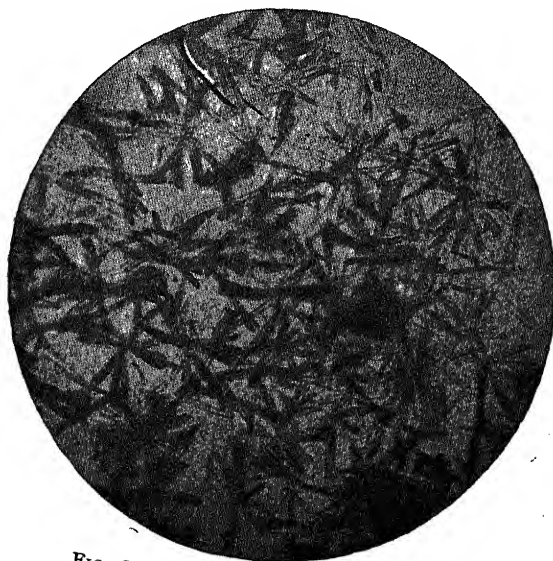


FIG. 61.—Hydrogenated fat $\times 50$ dia.

regulations on the part of the American authorities. Nevertheless, a considerable amount is still sent over, particularly to Germany.

The detection of this form of adulteration necessitates a very searching investigation. The fat most frequently added to lard is refined cotton-seed oil, with which is mixed suitable quantities of beef fat or beef stearin to compensate its fluidity and high iodine number. The use of other oils, in place of that from cotton-seed—that is, earth-nut, sesame, etc.—is naturally not precluded when the state of prices permit. A number of simple reagents have been proposed for the detection of such additions, among them being *alcoholic silver nitrate solution* (Pattinson test). Pure lard does not respond to this test, but not lard that has been prepared, even with the most scrupulous precaution, on a small scale. Although some commercial brands give a reaction with this test, one is not therefore justified in considering them as necessarily adulterated, since very minute quantities of extraneous substances are sufficient to produce discoloration of the silver nitrate, and such quantities are often present in the commercial grades of lard. The same applies to molybdic acid solution, this test, also, being so delicate that the majority of the commercial qualities of lard will not pass it. It is performed as follows: To 1 grm. of the suspected fat, dissolved in about 5 c.c. of chloroform, are added 2 or 3 c.c. of a solution of pure sodium phospho-molybdate, and the whole shaken up. After a few minutes' rest the two liquids will have separated again, and the reagent solution overlaying the chloroform will be found coloured a more or less intense green to greenish-blue, according to the percentage of reducing substances in the lard. The same applies equally to the so-called Hirschsohn reaction for detecting cotton-seed oil, of which it is said to reveal down to 5 per cent. To 5 grms. of the suspected fat are added 5 or 6 drops of a solution of 1 grm. of gold chloride in 200

grms. of chloroform, the mixture being shaken up and immersed in boiling water for twenty minutes. In presence of cotton-seed oil the liquid will appear rose-colour to red by reflected light and greenish by transmitted light.

Lead acetate has also been recommended as a reagent, and is said to produce a more or less orange-red coloration when ammonia is added to the agitated mixture of acetate and melted fats. These reactions are all, however, uncertain, and the only sure means of forming an accurate decision is by a comprehensive examination embracing :—

1. The microscopic appearance of the melted and re-crystallised fat ;
2. The melting-point ;
3. The saponification value ;
4. The melting- and setting-points of the fatty acids ;
5. The iodine number ;
6. The specific gravity.

In connection with the iodine number of lard, it should be mentioned that it does not always enable an accurate conclusion to be drawn, the reason for this being the variable proportion of olein and linolin present, the iodine number of which is much higher than that of the fat. Moreover, certain not unimportant deviations are exhibited in the lards obtained from various portions of the body of the hog. Thus Dietrich found lard from dripping to have an iodine number of 47.5 to 55.3 and that from bacon 60.0. In individual instances, however, the iodine number can be utilised in the determination of lard.

Dr. A. Goske investigated lard, particular regard being paid to the detection of tallow by crystallisation. The earlier experiments were performed on steam lard (American crude lard) and the so-called neutral lard (the edible lard) prepared therefrom, both of which can be readily examined for tallow by crystallisation. In the case of butchers' lard, other con-

ditions obtain, the crystals forming herein exhibiting marked differences compared with those from American lard. By dissolving the fat in ether and allowing it to re-crystallise, the crystals obtained in the absence of tallow are lamellar with oblique ends, whereas when tallow is present they take the form of aggregations of small curved needles. The result of the crystallisation is in a measure dependent on the temperature prevailing and on the amount of fat in the solution; the smaller the quantity of fat and the higher the temperature the better do the crystals come out. It is best to take not more than 1 gm. of lard, allow the tube to cool slowly in a beaker of water and to leave it to stand at 12°-13° C., since if the solution be set at lower temperatures (*e.g.*, 4° C. and under), good and characteristic crystals cannot be obtained. With oily lards—and in the summer time 2 c.c. of lard may be required—10 c.c. of ether is employed and the test tube loosely plugged with cotton wool.

When the crystallisation has been satisfactorily performed, the crystals of pure steam lard appear under the microscope as large sharply-defined plates, often up to $\frac{1}{4}$ mm. wide, their mass being here and there interspersed with aggregations of long needles, though slight modifications of this ordinary type may occur. Lard containing beef stearin crystallises in small fascicles of needles starting from a common centre, more or less curved, and in sheaves or brushes (Fig. 58). Latterly, oleomargarine has on occasion been employed as an adulterant, in which event the appearance of the crystals as a whole differs from that presented in the case of lard mixed with pressed stearin.

Butchers' tallow shows a remarkable difference in crystalline habit, in that no true lard stearin lamellae can be obtained therefrom, their presence being at most merely indicated. "Butchers' tallow crystallises in needles," which are considerably longer than tallow needles, frequently felted or fascicular

and occasionally resting on a common stalk. Goske's researches also aimed at elucidating the reason why American lard is richer in olein than butchers' lard, but failed to realise their object, although this has since been proved to be due to the difference in feeding, American hogs being fed largely on maize.

On recrystallising beef fat from ether several times it loses its characteristic appearance, gradually assuming the form of long thin plates, like those of lard but not so broad. On the other hand, the lard deposit after several crystallisations also forms in thinner crystals, so that the two cannot be distinguished. In fact, the products approximate to each other, forming a palmito-distearin with a melting-point of 62° (see Figs. 56 and 57).

With regard to the detection of cotton oil in lard, Goske abandoned the use of phospho-molybdate, since this reagent is reduced by any lard that is somewhat old. On the other hand, Becchi's reaction was found useful when performed in the following manner, fat that was as much as three or four months old, and had been left exposed and had thereby acquired a repellent smell, exhibiting no reducing action. In cotton-seed oil the reducing power appears to diminish with age. The reaction is performed in the manner recommended by the Italian Committee, the following solutions being used separately:—

(1) 1 grm. of silver nitrate dissolved in 200 c.c. of 98 per cent. alcohol, 40 c.c. of ether and 0.1 of nitric acid.

(2) 15 c.c. of colza oil in 100 c.c. of amyl alcohol.

Equal parts (5 c.c.) of fat and solution No. 2 with $\frac{1}{2}$ c.c. of solution No. 1 are heated for a quarter of an hour in a briskly boiling water bath.

For observing the reaction the test tube is held in front of a sheet of white paper. However, in doubtful cases, where no decided brown coloration is produced, one must not rest contented with the Becchi reaction alone.

Jean's method of testing lard and fatty oils is based both on the refractometer test for the liquid fatty acids of the product under examination and on the determination of their saponification value. The liquid fatty acids are isolated from the total fatty acids by conversion into zinc soaps (by agitating the carbon bisulphide solution with zinc oxide) and subsequently decomposing the compound. Lard mixed with vegetable oils can be recognised by its fatty acids producing a smaller deviation than -30° , the saponification value being at the same time below 190-191.

The subjoined figures were obtained by E. Späth in the examination of lard, beef tallow, and cotton-seed oil:—

	Iodine Number.	Acetyl Acid Number.	Acetyl Saponi- fication Value.	Acetyl Number
Lard, self-run	58.7-59.8	201.7-204.1	207.2-207.6	4.55-5.5
Older lard	52.0-52.4	198.9-199.2	201.1-205.2	5.1-6.0
Alimentary lard	63.8	196.0	205.7	9.7
Beef tallow, self-run . .	43.3	196.9-199.0	201.8-204.7	4.4-5.7
Cotton-seed oil, fresh . .	102.6	193.3-196.2	213.3-217.0	20.0-20.8
" " older	99.7	193.0	216.0	23.0
Mixture : 60 parts lard, 20 parts beef tallow and 20 parts cotton seed oil . .	62.0	190.6-192.1	200.2-204.3	10.4-12.2

Dr. Mansfeld says, with regard to the examination of lard: "The difficulties in testing the purity of lard reside, not merely in the manifold, and mostly complex, methods of falsification practised, but also in the great variations existing in the figures reported in the literature of the subject".

In testing for vegetable oils the Becchi and Welman's reactions are employed, but require a little care, since the reactions may be faintly manifested even by pure lard. Clear filtration of the fat is particularly essential. Since, in the case of complex adulterations, a fat may give a normal iodine number, these qualitative tests should be invariably performed. In such instances a more detailed examination of the fatty acids affords much information.

See also p. 304. A process differing somewhat from that hitherto employed for the preparation of the lead soaps, and one that is used in the laboratory of the Paris Bourse, is given below.

About 10 grms. of the solid fatty acids are dissolved in 100 c.c. of ether, and 3 grms. of zinc oxide are added, the whole being then well shaken up for some time: zinc soaps are formed, of which the oleates are soluble in ether. The solution is then filtered by the aid of an aspirator, the ether removed by distillation, and the residue mixed with 20 c.c. of hot dilute hydrochloric acid (1 : 4). Thereupon it is washed twice with hot water in a separating funnel, and filtered and dried as described above.

Pure lard gives the following figures:—

Iodine Number of the Oleic Acid.—About 92.

Refractometer Index.—44-45.

In the case of mixtures containing vegetable oils, both sets of figures are considerably increased in consequence of the presence of linolic and linolenic acids. (See pp. 110, 147 and 148).

Other tests which may be applied are the Halphen test and the nitric acid test. Halphen takes 1 or 2 c.c. of melted lard in a test tube, adds an equal volume of amyl alcohol, and the same of a 1 per cent. solution of sulphur in carbon bisulphide. If cotton-seed oil is present, a crimson colour will be developed within half an hour on heating in the water bath.

The nitric acid test consists in taking 1 or 2 c.c. of melted lard and shaking vigorously with an equal volume of nitric acid, specific gravity 1.375, in presence of cotton-seed oils, a chocolate brown coloration quickly develops.

It must be remarked, however, that in the purification of cotton-seed oil or on heating it to a high temperature, the constituents which yield the above reactions are more or less

destroyed, and the material thus treated either does not respond or only responds feebly to these reactions.

Sesame oil, which is sometimes used for adulterating lard, may be detected by the Baudouin reaction. This consists in shaking the melted lard with one or two c.c. of concentrated hydrochloric acid and a pinch of cane sugar, when in presence of sesame oil a pink colour is formed. In place of sugar, a few drops of a 1 per cent. solution of furfurol may be employed.

Earth-nut oil is detected by the presence of arachidic acid by the method recommended by Renard. The lard is saponified with alkali, the fatty acids separated by hydrochloric acid being dissolved in alcohol and precipitated as lead salts by adding lead acetate. The lead salts soluble in ether are separated by shaking with this liquid, and the insoluble lead salts decomposed by acid. The solid fatty acids thus obtained are dissolved in 90 per cent. alcohol and set aside to crystallise. The crystals which separate are washed with alcohol and their melting-point determined. Arachidic acid has a melting-point of 74° to 75.5° . By taking a weighed quantity of the fat and also weighing the arachidic acid an approximate idea of the proportion of earth-nut oil present can be formed. Earth-nut oil itself was found to yield about 5 per cent. of arachidic acid.

Detection of Paraffin Wax.—About 5 grams of the fat is saponified by heating with 1.5 grams of caustic potash and a little alcohol, the alcohol is evaporated off and the soap dissolved in warm water. On cooling, the liquid will be turbid if paraffin wax is present, otherwise, it is quite clear and transparent.

On heating the liquid some of the wax rises to the surface in minute spherules which, on cooling, show a characteristic striated surface. Paraffin wax will be found in the unsaponifiable matter which will therefore be greater in amount than usual.

Uses.—Medicinal: for ointments; cosmetic: for pomades, creams, etc.; alimantal purposes: as edible fat, and for cooking and frying: soap-making.

LARD OIL.

By pressing lard at zero C. lard oil, which does not set even at comparatively low temperatures, is obtained, leaving behind lard stearin, consisting of palmitin and stearin. Lard oil is thinly fluid like olive oil, pale yellow, and still contains (according to the method used and care exercised in its preparation) very variable quantities of palmitin and stearin, on which account its specific gravity and setting-point fluctuate considerably. Commercial lard oils have the following constants:

Specific gravity at 15° C.	0.916
Saponification number	189.9-194.0
Iodine number	67.0-88.0
Refractive index at 40°	1.4511

The free acid is usually very small.

The constants of lard stearin are as follows:—

Specific gravity at 35° C.	.9031
Melting-point	44.3°
Saponification number	194.5-195.5
Iodine number	44.24-54.3
Refractive index at 40°	1.4576-1.4584

This oil finds employment as an edible oil and for lubricating purposes, as well as extensive use for wool-softening, replacing olive oil and spermaceti owing to its lower price, and being also used in France, England, and America for adulterating these oils. The solid portion left behind in the hydraulic press forms a very suitable material for candle-making, and as lard stearin is used for stiffening fluid lards coming (like lard oil) principally from America (Cincinnati), it finds a market in England, France, and Germany.

BEEF MARROW.

Raw Material.—The large hollow bones of the ox contain a fat or marrow which is similar to tallow, and is indebted for its characteristic taste to the cellular tissues which accompany it.

Preparation.—The bones are opened, *i.e.*, split or sawn, whilst still fresh, the marrow from old bones has no especial value, and is then only classed as bone fat. It is carefully melted, separated from the cell tissue by straining, and stored in a suitable manner.

Constants of beef marrow :—

Specific gravity at 15°	0.9311-0.938
Solidifying point	29°-31°
Melting-point	37°-43°
Acid number	0.44-1.6
Saponification number	195.8-199.6
Iodine number	39.2-55.4
Refractive index at 25°	1.4628

Uses.—For alimentary purposes; as an addition to soups, which it strengthens; in cosmetics: for pomades, ointments, etc.

B.—USED IN INDUSTRIAL PROCESSES.

TALLOW, BEEF TALLOW (TALG, RINDTALG, OCHSEN-TALG, UNSCHLITT).

Raw Material.—The fat of ruminating animals (oxen, cows, steers, calves), which is enclosed in the cellular tissue and is still commingled with particles of skin or blood in larger or smaller quantities. As soon as the beast has been slaughtered, flayed and cut up, the fat is sorted into crude prime (Rohkern) and crude cuttings (Rohausschnitt). The former, the kidney fat, comprises the greater coherent fat masses which are designated, according to their position in the body, as intestinal fat, heart fat, lung fat, neck fat, purse fat

(from the scrotum), and kidney fat, and is almost pure fat, free from particles of blood and flesh.

To the second category belong the waste fats, copiously interspersed with portions of blood and skin, from the limbs, etc. These crude cuttings come into the melter's hands in a comparatively fresh state, whereas the waste pieces separated from the meat in trimming are collected slowly and sometimes have commenced to putrify, owing to the length of time they have been saved up to make a respectable quantity for delivery to the melter. If only a little more care were taken in storing the fat, by hanging the pieces up to cool and dry separately instead of throwing them into a heap whilst still warm, the very rapid putrefaction of the particles of blood, flesh and cellular tissue would be prevented and the fat-melting process no longer attended with such malodorous exhalations.

Also in the tallow-melting works, large parcels which cannot be worked up at once could be preserved, by suitable cold storage and salting, to postpone if even for a few days, the decomposition of the nitrogenous matters. For the preservation of crude tallow destined for alimentary purposes, Schaedler proposes the following method: The fat is placed in vats containing water and 3 to 4 per cent. of leaven and is there left, the leaven undergoing continual decomposition and thereby protecting the fat from atmospheric oxygen. The acids—acetic and lactic acid—formed dissolve the animal membrane and expose the fat, so that the residue left behind in the ensuing process of melting is but small. The residual water must be treated like manure heap drainings.

Preparing the Tallow.—By melting the crude tallow and pressing the residual cellular tissue, particles of flesh, etc.

For the rational utilization of the crude tallow it must be reduced to small pieces, because the fat is not merely mixed

with tissue but also enclosed in cell capsules; by this treatment the capsules are ruptured and the outflow of the fat facilitated. The comminution—which is mostly into small cubes—is effected either on wooden blocks with a straight-edged chopper or in a kind of stamping trough with a pestle or pounder having an S-shaped cutting edge, an arrangement such as is sometimes used for cutting up roots for fodder. Another form is a knife worked on a hinge with one hand, whilst the fat is laid on the block with the other. In England edge runners are used for crushing and breaking down the cellular tissue. Fat cutters with rotating knives (see Figs. 3 and 4) are also suited for performing the operation by machinery.

Three different methods are pursued in melting out the fat:—

1. The ordinary melting in an open pan over direct fire (the so-called greaves method or dry rendering);
2. Melting with dilute sulphuric acid over direct fire (wet rendering);
3. Melting by steam or in a closed apparatus of some kind.

As in the case of other factories, tallow-melting works are subject to certain official sanitary regulations, which either prohibit the work being carried on in inhabited districts or permit it only under restrictions chiefly concerned with the suppression of the insupportable exhalations attendant on this industry. Melting over direct fire is now confined to small works, larger factories employing only the methods specified under 2 and 3.

1. Melting in Open Pans over Direct Fire (Rendering).

In this, the oldest method of all, a sheet-iron or copper pan is set in brickwork so that the flame impinges on the bottom only, since otherwise the fatty mass would become too

strongly heated. The crude fat, along with a small proportion of water, is placed in the pan and the fire kindled; the fat soon begins to run and finally forms a liquid rendered somewhat milky in appearance by the water initially present in the tallow and that added thereto. The expansion of the fat, in liquefying, ruptures the cellular envelopes, which then, under the influence of the continual heat and the evaporation of the water, contract and form, as it were, crumbs (the greaves) floating about in the fatty mass, the latter by degrees becoming clear. According to the size of the pan and the strength of the fire, as well as the amount of water present, the operation lasts from one to several hours, the mass having to be continually stirred with wooden paddles to prevent the residual tissue subsiding to the bottom of the pan and becoming carbonised. When the fat flows quietly and seems clear the pan is emptied and the greaves separated, to which end a strong sieve of sheet-copper of a diameter corresponding with that of the pan is placed in the latter and pressed downwards by means of the handles, so that the liquid fat rises through the perforations whilst the greaves are forced to the bottom. The tallow is then ladled out or run off, through a tap at a convenient height at the side of the pan, into a wooden vat or other suitable vessel covered with a filter cloth stretched over a frame or laid on a perforated plate, so that any residual particles of tissue still in the fat are retained, and the tallow freed from all solid matter. If the tallow is to be refined at once it can be run off into a second pan standing at a lower level.

In the melting pan there remain the greaves with some adherent fat—it being impossible to remove all the fat, however well the copper sieve may have been forced into the mass—and these are now subjected to pressure, the contents of the pan being filled into press-bags of woollen or horse-hair cloth, which are then placed in the greaves press. One of the

oldest forms of press employed for this purpose is the screw press shown in Fig. 62. In this the press-bag containing greaves is placed in the cylinder D, which is made in two halves connected by screws, and consists of plaited strong iron wire or perforated plate strengthened by iron struts. It is open at both ends and stands in a sheet-iron trough A fitted

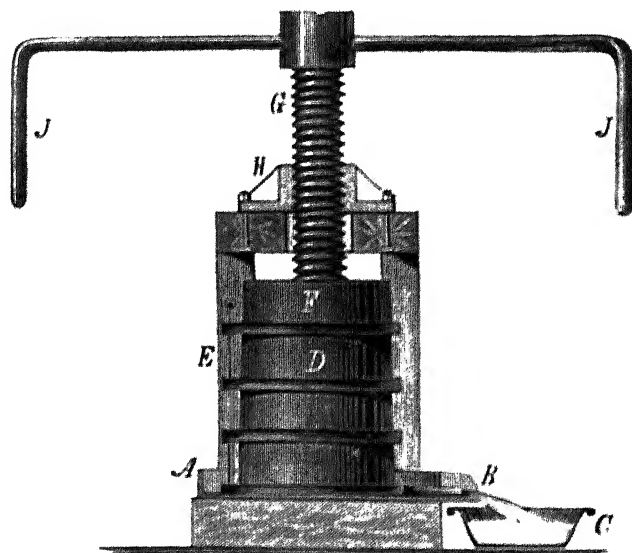


FIG. 62. Press for greaves.—A, sheet iron trough; B, discharge pipe; C, collecting dish; D, cylinder; E, frame; F, press head; G, screw spindle; H, screw socket; J, lever.

with an outflow nozzle B. At the upper end the press head F fits accurately in the cylinder and is moved up and down by the strong screw spindle G working in the socket H on the cross beams of the press frame and driven by the lever J, thus exerting a strong pressure on the greaves and expressing the fat.

Improved presses for greaves are displayed in Figs. 63 and 64, that with ratchet lever gear furnishing very good results.

Other presses, notably hydraulic presses, may, of course, be also used for the same purpose.

The greaves presses are always placed near the melting pans or in some other warm situation, in order to prevent the tallow from setting. The yield obtained by melting crude fat amounts to 80-82 per cent. of tallow and 10-15 per cent. of

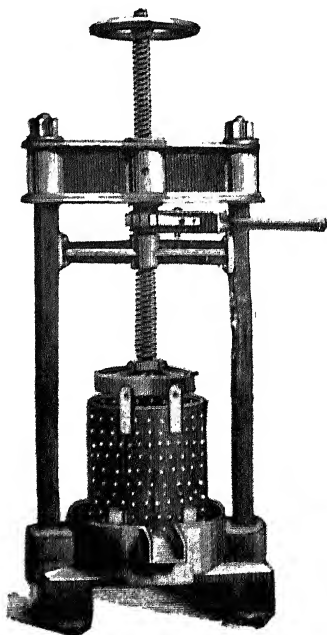


FIG. 63.—Greaves press with ratchet lever gear.

residue (greaves); very pure and dry crude fats, such as kidney suet, yield 90 per cent. and upwards of pure tallow. The loaf or cake tallow is obtained from the melted tallow ladled out of the pan and poured into wooden vessels, the yellow coloured product from the pressing of the greaves forming cask tallow.

Occasionally a weak brine is added to the tallow in the melting pan, its action being to allow a higher temperature in

the mass, and a shrinkage of the cellular tissues. The pure white tallow is known as "virgin tallow," that from the press being "greave tallow" or "second runnings".

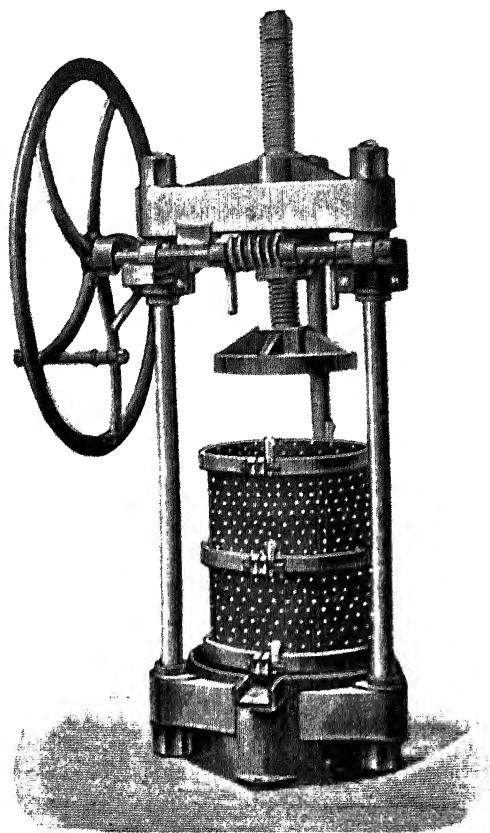


FIG. 64.—Greaves press.

The greaves, even when most carefully pressed, still retain up to 10 per cent. of fat. Formerly the very brown or carbonised greaves were much used as fuel and the better kinds given to pigs and poultry; but in large works, where they are produced in quantity, they are now extracted by car-

bon bisulphide or benzine, etc., and the product employed as a lubricant. The residual greaves, which, when no carbonisation has occurred in the melting process, consist of pure cellular tissue, may be worked up for the manufacture of glue, ferrocyanide, ammonia, manure, or nitrogenous charcoal.

As already mentioned, malodorous vapours and gases are evolved into the air during the melting of tallow in open pans and infect the neighbourhood. These gases and vapours contain, in addition to volatile acids (acetic and butyric acids), the constituents of the empyreumatic animal oils, such as ammonium cyanide, ammonium sulphide, sulphuretted hydrogen, pyridin, picolin, lutidin, etc. Acrolein vapours should not, however, occur, their formation taking place at a higher temperature than is necessary for melting tallow.

The inconveniences resulting from the open pan melting of crude tallow can be obviated by means of suitable contrivances. These comprise a tight-fitting pan lid provided with a flue pipe, through which the vapours can be drawn into the smoke stack and discharged into the air at higher levels.

A stirring apparatus, worked from the outside, must, of course, in these cases be employed. The gases can also be exhausted direct by a current of air. Appliances constructed on these or similar principles are supplied by Thomson, Errard, D'Arcet, Wilson, Buff, Foulé and others.

According to Schaedler, the process of melting along with caustic soda is greatly under-estimated, notwithstanding that the malodorous fatty acids are, by this method, brought into combination. The temperature need not exceed 100°C ., 100 parts of fat being mixed with an equal weight of a 1 to $1\frac{1}{2}$ per cent. caustic soda lye and heated by a current of steam. The cellular tissue is greatly distended by the alkali, and the floating fat, after being kept in a liquid condition for a few hours to clarify, is washed with water, 95 per cent. of an inodorous fat being obtainable by this means. The alkaline water when

subsequently treated with acids gives off an odour of the volatile fatty acids derived from the original fat. This method is, however, unsuitable for very old fats, ammonia being in such cases evolved. The mass also froths up very considerably when heated and the separation of the fat from the emulsion of lye is attended with much difficulty.

2. Melting with Dilute Sulphuric Acid.

D'Arcet prefers to melt tallow along with dilute sulphuric acid, 100 parts of tallow being, as a rule, employed with 50 of water and 1 of sulphuric acid (specific gravity, 1.848). When the mixture has been heated during several hours, to such a degree that the dilute acid boils continuously, the whole of the fat will be gradually obtained floating on the surface, because the cells of the fatty tissue are, for the most part, destroyed, and the fat liberated. On examining the greaves under the microscope it will be found that the still unbroken cells no longer contain fat, but are filled with dilute sulphuric acid, which has penetrated into the interior and expelled the fat therefrom.

D'Arcet's process presents the advantage of recovering the *whole* of the fat without the necessity of pressing the greaves, since the latter merely contain dilute sulphuric acid. An inconvenience, however, attends this process in that the resulting greaves can no longer be exclusively employed as fodder, though it is erroneous to assert that they are altogether unfit for that purpose, since numerous experiments have demonstrated that such acid greaves, when mixed with other food stuffs, can very well be used for feeding pigs without the latter losing their appetite or the power of laying on flesh. The chief drawback to the D'Arcet process is that iron vessels, being rapidly corroded by the sulphuric acid, cannot be used, and recourse must be had to materials (*e.g.*, lead) unattacked or only inappreciably affected by this acid. To avoid making the vessels too heavy, as they would be if constructed of lead

alone, iron pans are lined internally with sheet lead a few millimetres in thickness, the joints of the plates being of course soldered autogeneously by pure molten lead. When steam is available the treatment of tallow by the sulphuric acid process can be carried on in lead-lined wooden vats, or when wooden vessels are easily procurable the leaden lining may be omitted, since these vessels, especially when made of woods rich in resinous matter, can be used for a comparatively long time.

Stein's Tallow-Melting Process. According to Stein, the pans serving for melting the tallow should be fitted with a grated false bottom to support the greaves and prevent their charring, the employment of stirrers thus becoming unnecessary. The pan is fitted with a lid perforated and covered with a linen cloth supporting a layer (3-3½ inches thick) of alternating strata of lime and charcoal, through which the evolved gases ascend and are thereby deodorised.

This disinfecting layer must be renewed each time the pan is recharged, a troublesome task and one disturbing the regularity of the work. Burning the vapours always affords the most satisfactory solution.

Renard's Tallow-Melting Process.—Renard employs for the destruction of the cellular tissue a substance, *viz.*, caustic soda, which acts even more energetically than sulphuric acid on organic substances. As is well known, caustic soda in common with the other caustic alkalis has the power of dissolving animal integuments with ease. Since, however, the alkalis also possess the power of saponifying fats, a considerable loss of fatty matter would ensue, from saponification, were a large quantity of caustic soda employed, soaps soluble in the water used for melting being formed; but if the caustic alkali be employed merely in small proportion the saponification of the tallow need not be feared, very dilute solutions of alkali attacking the fat only after somewhat prolonged boiling,

whilst the complete destruction of the cell capsules is accomplished much quicker.

The use of caustic soda is undoubtedly preferable to sulphuric acid in tallow melting, the yield of tallow obtained thereby being the highest possible. A drawback—though not one to be greatly emphasised—to this method is that the greaves are lost, and the liquor containing the decomposed cellular substance cannot be used for any other purpose than the watering of dung heaps.

The process, as originated by Renard, contemplated the use of alkali carbonates, but there are many reasons for preferring to employ the alkali in the caustic condition, since though the carbonates have, it is true, the power of destroying the cellular tissue, the operation is a much more protracted one than in the case of caustic alkalis. Moreover, the use of carbonates in nowise precludes loss of fatty matter, the solution of these salts being capable of emulsifying the fat and dividing it into extremely fine globules, thus forming a milky liquid.

The quantity of caustic soda to be used in this process is relatively minute, 1 part dissolved in 100-200 parts of water being generally quite sufficient for 1000 parts of tallow. Only when the tallow under treatment contains a somewhat large proportion of cellular tissue may a rather larger quantity of caustic soda be taken; otherwise the boiling must be carried on for a longer time.

3. *Melting by Steam.*

Naturally, the melting of crude tallow by the aid of steam in closed vessels offers the greatest security for inodorous working and most complete utilization of the materials, on which account this method cannot be too warmly recommended. Various appliances have been constructed for this object, notably those of Gellhorn, Flottmann & Co., Lock-

wood & Everitt, Rivoir, Heintschel and others, as already illustrated and fully described in an earlier section of the present work.

Properties of Tallow.—Beef tallow is hard and solid, pale yellow or white, tasteless and inodorous in the fresh state, but assuming after a short time a rather unpleasant characteristic smell, without, however, becoming rancid. It is insoluble in cold alcohol but dissolves in boiling alcohol of specific gravity 0·822, as also in ether, chloroform, warm benzene and ethereal oils. The elementary composition is :—

Carbon	76·50 per cent.
Hydrogen	11·91 "
Oxygen	11·59 "
<hr/>	
	100·00

Specific gravity at 15° C.	0·943-0·952
" " 40° C.	0·895
Solidifying point	27°-35°
Melting-point	42°-48·5°
Saponification number	193·2-200
Iodine number	35·4-47·5
Refractive index at 40° C.	1·4596
Hegner value	95·4-96
Fatty acids—	
Specific gravity at 100° C.	0·8698
Solidifying point	43°-45°
Melting-point	43°-47°
Neutralization value	197·2-201·6
Iodine number	25·9-41·3
Refractive Index at 60° C.	1·4375

Beef tallow consists almost entirely of palmitin, stearin and olein; the percentage of the latter can be calculated from the iodine number. According to the parts of the body from whence the tallow is derived, the proportions of olein, stearin and palmitin vary, the softest and richest in olein being the pure fat from the scrotum; the richest in stearin, and consequently the hardest, that from the intestines. Schaedler gives the average combined percentage of stearin and palmitin as 66½ per cent.

MUTTON TALLOW (HAMMILLTALLO).

Raw Material.—The accumulations of fat in the flesh and tissues of the sheep.

Preparation.—Like beef tallow, by melting and by pressing the residual tissues from the melting process.

Properties. Mutton tallow is similar to beef tallow but is less highly coloured, being whiter, rather harder and more brittle. Initially inodorous it assumes, after a brief exposure to the air, the well-known characteristic smell and taste of mutton, very quickly becomes rancid and agrees in this property with goat's fat, which it then resembles in smell. These odours are due, according to Chevreul, to a volatile fatty acid (hircinic acid), which is, however, merely a mixture of butyric acid with other volatile fatty acids.

The composition of mutton tallow is very similar to beef tallow, the constants being given below:—

Specific gravity at 15° C.	0.937-0.953
Solidifying point	32°-41°
Melting-point	44°-49°
Saponification number	192-195.2
Iodine number	32.7-46.2
Hehner value	95.54
Refractive index at 60° C.	1.4504
Fatty acids—	
Solidifying point	39°-41°
Melting-point	46°-54°
Neutralization number	198
Iodine number	34.8
Refractive index at 60° C.	1.4374

Mutton tallow is soluble only in over 60 volumes of cold ether or 45 volumes of boiling alcohol of specific gravity 0.821.

It contains per 100 parts—

Carbon	76.61 per cent.
Hydrogen	12.03 "
Oxygen	11.36 "
	<hr/>
	100.00 "

and consists of about 22 per cent. of stearin, 31 per cent. of palmitin and 47 per cent. of olein.

The olein obtained by pressure is colourless, with a faint odour of mutton and a sp. gr. of 0.913 at 15° C.; 80 parts are dissolved by 100 parts of boiling absolute alcohol.

Iodine Value.—92.

On account of the crystalline appearance of the stearin yielded by this fat, mutton tallow is preferred by candle-makers to that of the ox; very frequently the two are met with melted together.

Uses.—Similar to those of beef tallow.

TALLOW OIL.

Olein or tallow oil (talgol) is obtained from beef tallow, as also from the other tallowy fats—mutton fat and goat fat—by seeding and pressure.

This tallow oil is somewhat thick and pulpy, and is highly suitable for the manufacture of fine soaps, provided the colour of the commercial article is good; it is also used in lubricating machinery. The press residue forms the ordinary stearin of commerce. In order to deprive the tallow of its entire content of olein, it is melted, then stirred continually until nearly cold. The mass is left to cool in flat tin moulds, the cakes wrapped in press cloths and pressed between iron plates in the hydraulic press, whereby the olein is forced out, leaving the stearin and palmitin behind.

The value of the olein is greater in inverse proportion to the amount of solid fats it contains.

There is a distinction between tallow oil (olein) and oleo-margarine (styled "oleo"), the latter being prepared from fresh unmelted beef kidney fat by pressure, and used either direct as an edible fat or mixed with oil and milk and worked up into artificial butter (margarine).

Tallow oil is a liquid solidifying in winter time, of a

specific gravity of 0.94 at 15° C. It is much more readily soluble than palmitin or stearin in absolute alcohol, but it is insoluble in dilute alcohol. In the same way that oleic acid is converted by the action of nitrous acid into elaidic acid, so also olein, under the same conditions, passes over into a solid isomeric state, forming elaidin, consisting of crystalline nodules which melt at 32° C. according to Mayer, or 38° C. according to Duffy, and dissolve readily in ether, though almost insoluble in alcohol.

(Oleomargarine has the following constants:—

Specific gravity at 15° C.	0.924-0.930
Melting-point	17°3'-26°3'
Saponification number	195.0-197.4
Free fatty acids	0.56-1.56
Iodine number	44.5-53.3
Hehner "	95.56
Reichert "	0.4-0.6

Fatty acids:—

Melting-point	42°0'
Solidifying point	39°8'

and consists of:—

Triolein	50.8-55.2
Tripalmitin	15.6-40.0
Tristearin	68.3-39.9

The commercial grades are: Melted tallow, press tallow or stearin, and margarine.

The constants of beef stearin are as follows:—

Specific gravity at 35° C.	0.906
Melting-point	49°-56°
Solidifying point	41°5'-43°5'
Saponification number	194-196
Iodine number	8-17
Refractive index at 25° C.	1.4576-1.4584

In the Paris market tallow is sold on the "titer" or setting-point of the mixed fatty acids.

According to De Scheepper and Geitel, tallow and similar fats, etc., have the subjoined setting-points: -

Tallow	40°46' C.
Oleomargarine	38°14' "
Stearin	50°5' "
Mutton tallow	46°1' "
Beef tallow	44°5' "
Bone fat	40°7'-42°3' "
Olive oil	40°05' "
Cotton-seed oil	34°0' "
Cocoa-nut oil	23°0' "
Stearin grease	44°0' "

"*Suif d'épluchures*" (a French commercial brand which, when dried, smells of sulphurous acid, is green in colour and is probably a mixture of animal fat with inferior olive oil).

Adulterations of Tallow. - Beef tallow is mostly adulterated with fats of low value, such as fish tallow (whale fat), bone fat, pot fat, and latterly with distilled wool fat. According to Schaedler, such falsification cannot be considered as fraudulent, provided the product is not sold under the general designation of tallow; the addition of these fats serves to lower the quality of the product, certainly, but the manufacturer can supply such mixtures at lower rates for industries where purity is of no importance. Adulteration of tallow with cheap fatty acids, notably those of wool fat, is indicated by the smell, which cannot be completely removed during the distillation of wool fat.

Tallows falsified with fish tallow have their melting-point lowered considerably, and can be recognised in a superficial manner by the smell of fish oil evolved on gently melting the fat along with common salt. There are other adulterations practised by the addition of starch, potato flour or mineral substances, which can be identified, as in the case of lard; while cotton-seed oil is also used as an adulterant when the price permits.

EXAMINATION OF TALLOW.

Estimation of Moisture.—5 grms. of the fat are placed in a tared glass beaker or basin containing a glass rod and dried, till constant, at 105° C., with constant stirring to prevent bumping. If tallow be mixed with potash its absorptive capacity for water is increased. In such event the fat cannot be freed from water by drying at 100° C., the best plan then being to determine the content of fatty matter, impurities and potash, and estimate moisture by difference.

Non-fats (solid matters, fragments of skin, vegetable matter, dirt, etc.).—10-20 grms. of fat are extracted in a flask with chloroform or benzol and then poured through a previously tared dry filter, washing with the same solvent until a drop of the liquid allowed to fall on paper leaves no grease spot. The solution is then dried at 100° C. and weighed. If on incinerating the residue a large amount of ash is left, the sample has been mixed with some inorganic substance, chalk, clay, etc., the nature of which can be ascertained by the ordinary processes of qualitative analysis. When a copious organic residue is left after extraction, it is tested by moistening with iodine solution. A blue coloration indicates starchy matter (starch, flour or potato pulp), the presence of which is also detectable by microscopic examination. Moreover, 1 part of the suspected fat may be boiled with 2 parts of acidified water in a beaker for a few moments and then placed in water at 40° C., so that the fat does not set too quickly for the impurities to subside. If iodine tincture be then added, a blue coloration is immediately developed. The starchy matters are freed only with some difficulty from fat by extraction, so that the weight of the dried residue after deduction of the ash does not exactly correspond to the amount of starch present.

Substances soluble in water are often left behind after the extraction with chloroform and can be identified (e.g.,

salt) by an analysis of the residue, or else they may be removed from the fat by taking a larger quantity of the fat, say 50 to 100 grams—and shaking it up in warm water to melt the solid fats, the mixture being then left at rest for a day or two to allow of a complete separation into the two layers (fat and water) is effected. If this does not ensue, even after prolonged resting, the fat remains partly in the form of an emulsion, but the fat particles may be united by shaking up with ether; the ether being then removed by the aid of a separating funnel and tested.

Sulphuric acid, remaining from the refining process, will be found in the aqueous extract, and may be estimated by adding caustic soda, using methylorange as indicator. After removing other dissolved substances the solution is evaporated to dryness and the residue examined.

Essential oils, in admixture with the fat, are removed by distillation and determined by the loss in weight. The distillate may be shaken up with ether, the latter evaporated and the residue examined qualitatively.

In unadulterated tallows the non-fats consist mainly of impurities of skin and gelatine; in bone tallows, calcium phosphate may be present.

Artificially soft tallow from cuttings is found to have been adulterated with lime which was stirred in whilst the fat was in the liquid state, the result being a hardening of the tallow owing to the formation of a lime soap. In the ether extraction for the determination of non-fatty matter this admixture is left in the residue, which must then be dried for its estimation.

Examination of the dried and filtered fat is not confined merely to the detection of adulteration, but is also performed with the object of determining the value of the article.

Determining the Value of Tallow.—According to Dalican, the value of the tallow increases with its melting-point.

Wolkenhaar considers that tallow having a melting point of less than 40° C. is inadmissible, whereas Dalican, who, with greater correctness, determines the value by the setting-point of the fatty acids (tallow "titer"), gives 44° C. as the lowest limit therefor. The determination of the melting-point yields results varying considerably, according to the way the test is performed, it appears therefore to be more advantageous to determine the melting- and setting-points of the fatty acids obtained from the fat.

The following table by Dalican is used in candle works to calculate the yield of solid acids (commercial stearic + stearic and palmitic) and oleine (crude oleic acid) obtainable from tallows of various melting-points :—

Setting-Point.	Stearic Acid. Per Cent.	Oleic Acid Per Cent.
35	25.20	69.80
37	29.80	65.20
39	33.45	61.55
41	38.00	57.00
43	43.70	51.30
45	51.30	43.70
47	57.95	37.05
49	71.25	23.75
51	79.50	15.50
53	92.10	2.90

The constitution of the tallow can be calculated from the iodine number, either the fat itself or the fatty acids therefrom being titrated. Theoretically, the iodine number of pure oleic acid is 90.07, that obtained experimentally being concordantly between 89.8 and 90.5. The iodine number of olein is 86.20. The iodine number of a fat being found, and expressed as i , then the olein percentage content O and the amount of oleic acid obtainable therefrom, E , can be calculated as follows :—

$$O = \frac{100}{86.20} i \quad E = \frac{100}{90.07} i$$

or

$$O = 1.160 i \quad E = 1.1102 i$$

If i be the iodine number of the liquid fatty acids, then their acid content is also—

$$E = 1.1102 i$$

A high percentage of the liquid fatty acids lowers the value of the tallow for making stearic acids owing to the lower yield of solid acids and the dark colour of these acids when produced by the lime saponification process.

Detection of Fatty Adulterations.—The fatty adulterants of tallow are: paraffin, palm-kernel oil, coco-nut oil, cotton-seed stearin, wool fat, mutton tallow, rosin and rosin oils, fish tallow and bone fat.

FREE FATTY ACIDS IN BEEF TALLOW.

Kind of Tallow.	No. of Samples.	Acid Value.	Per Cent.	Remarks.
Russian	5	5.1 24.6	2.55 12.3	Old
"	2	10.1 12.4	5.05 6.2	Fresher
" P.Y.C.	1	4.3 10.4	2.2 5.2	Old
" "	3	4.3 4.7	2.2 2.35	Fresh
Australian	4	3.5 30.47	1.75 15.2	
Town tallow	2	9.0 14.2	4.5 7.1	
" "	1	50	35	6 years old

TITRE TEST OF TALLOW.

The accompanying table by Dalican and Jean shows the titer test of several different grades of tallow and admixtures thereof:

Nature of Tallow.	Titre, Degree C.
Paris town	43.5
Ordinary beef	44.0
Beef, kidney pure	45.5
Mutton, ordinary	46.0
" kidney	48.0
Bone fat	42.5
Intestinal tallow	41.0
St. Petersburg P.Y.C.	43.5
Odessa beef	44.5
" mutton	45.0
New York Prime City	44.0
" " Butchers' Association	44.5

Nature of Tallow		Temp. Degree C.
Western (Chicago)		45°0
Buenos Ayres beef		45°0
" " mutton		44°4
Australian mutton		44°8
" beef and mutton		44°5
" beef		43°5
Florence		44°5
Vienna		44°5
Goat		44°40
Horse fat		47°20
" " white		46°7
Surt d's pluchures		44°2
Green fat, souffrice		44°4
" " fatty acids		44°5
Distilled grease, souffrice		44°6
Slaughter-house, pure		45°4
Caul fat, pure		44°45
Mutton tallow, pure		48°4
25 mutton 75 slaughter-house		46°15
80 " 20 "		47°8
33°4 " 66°6 "		46°4
40°0 " 60°0 "		46°6
50°0 " 50°0 "		46°0
40°0 " 60°0 "		46°6
75°0 " 25°0 "		47°65
60°0 " 40°0 "		47°4
80°0 " 20°0 "		47°8
80°0 " 20°0 "		47°8
40 slaughter-house, 60 caul fat		44°65
33°4 " 66 "		44°45
80 " 20 "		44°8
75 " 25 "		44°65
Commercial stearic acid (stearic and palmitic), Renner		44°9
" " " " " Perré		54°8
" " " " " Chézy		53°0

The composition of mixtures of palmitic and stearic acids cannot be determined by the melting- or solidifying-points as shown in the following figures by Carlinfonti and Levi-Malvano and by Heintz :—

Percentage of		Solidifying point.	Solidifying point.	Melting-point.
Stearic Acid.	Palmitic Acid.	Carlinfonti and Levi-Malyano.	Heintz.	Heintz.
		° C.	° C.	° C.
100	0	68.2	—	69.2
90	10	65.90	62.5	67.2
80	20	63.50	60.3	65.3
70	30	60.80	59.3	62.9
60	40	57.65	56.5	60.3
50	50	56.25	55.0	56.6
40	60	55.90	54.5	56.3
30	70	54.75	54.0	55.2
32.5	67.5		54.0	55.1
20	80	55.75	53.8	57.5
10	90	58.40	51.5	60.1
0	100	61.0		62

The two tables appended are also by the former authors :—

Percentage of		Solidifying point.	Percentage of		Solidifying point.
Stearic Acid.	Oleic Acid.		Palmitic Acid.	Oleic Acid.	
		° C.			° C.
0	100	9	100	0	61
5	95	23.45	90	10	59.2
15	85	34.25	80	20	57.3
25	75	46.6	70	30	55.1
35	65	51.9	60	40	52.6
46	54	55.95	50	50	49.75
55	45	58.65	40	60	46.25
65	35	61.25	30	70	41.60
75	25	63.40	20	80	35
85	15	65.40	10	90	24.8
95	5	67.15	0	100	9
100	0	68.2	—	—	—

Coco-nut Oil and Palm-Kernel Oil.—The presence of these solid vegetable fats in tallow is revealed by the greatly increased saponification value, which is, for tallow, about 196, but for coco-nut oil 246-260 and for palm-kernel oil 242-250, also by the Reichert-Meissl number and Polenska numbers, see pp 116-117.

According to Rödiger these fats may be recognised in

tallow by the property exhibited by their soaps of salting out with greater difficulty than the tallow soaps. The soda lye for saponification is prepared by mixing 75 grams of caustic soda of precisely 38° Bé. at 15° C. with 150 grams of water, 150 grams of tallow are heated to boiling along with 50 grams of water in a tared basin and emulsified with 10 grams of soda lye, further additions of the latter being deferred until the first addition has been absorbed by the fat, which can be ascertained by taking a small sample out of the centre of the boiling soap solution with the wooden spatula employed for stirring and allowing it to run from the blade. If the mass breaks raggedly or draws out in viscous threads (which will be the case after further additions of lye), then the alkali has been absorbed, whilst on the contrary event a clear watery liquid will drip from the spatula after the turbid mass has run off. If this appearance does not alter, even after prolonged boiling, more water is added. When the operator is inexperienced he should continue the boiling for at least half an hour after 225 c.c. of lye have been used up (which is always the case), replacing, from time to time, the water lost by evaporation. The mass will then run off in a fairly viscous condition, and water must be added to make up the total initial volume of 425 c.c., 38° Bé. soda lye being afterwards added drop by drop from a weighed flaskful until a clear drop of lye appears on the dropping sample of soap. This occurs when the following additional quantities of lye have been added:—

Viennese beef tallow	25	grams of lye
Beef tallow with 5 per cent. of palm-kernel oil						29	" "
" " 10	"	"	"	"	"	37	" "
" " 15	"	"	"	"	"	42	" "
" " 20	"	"	"	"	"	45.5	" "

as low as 5 per cent. of palm-kernel oil can be detected

Distilled Wool Fat.—To detect this fat, which, according to Leopold Mayer, is employed to adulterate tallow, the sample is saponified and shaken up with ether, the residue left after evaporating the ether being tested for cholesterin (see p. 301). As no other fat contains such a large proportion of cholesterin, this test is perfectly reliable. There are no tri-glycerides in distilled wool fat, only free fatty acids, and, consequently, tallow adulterated with this fat will have a very high free fatty acid content. The fatty acids isolated from such a sample will become yellow in a few days' time and exhibit the characteristic smell of wool fat, due to the presence of volatile fatty acids.

Cotton Oil or Cotton-Seed Stearin.—Tallow falsified with cotton-seed oil or the stearin obtained therefrom will assume a red to red-brown coloration on treating 5 grams of the melted sample with 15 drops of nitric acid (specific gravity, 1.380) and shaking up briskly. On the other hand, pure beef or mutton tallow under this treatment will remain pure white, and palm-kernel oil and other fats show only a yellow coloration. According to Wolkenhaar this reaction is unreliable, since pure, but carelessly melted, samples of tallow behave similarly, and the test is only to some extent definite when the melting-point of the sample is below 40° C., and at the same time the specific gravity above 0.861 at 100°. It would be advisable to carefully filter such impure tallows, in order to remove the staining impurities before applying the test.

Cracan remarks in his treatise, that in the ordinary methods of testing beef tallow the quantitative reactions are not always sufficient, since it is easily possible by suitable mixtures to arbitrarily produce an article exhibiting the constants relied on as indicating purity. He elaborated a few reactions specially for the detection of Japan wax and for vegetable waxes in general, in respect of which he opined that adulterations of tallow therewith were not infrequent, such

falsifications being, however, scarcely to be apprehended in the present low prices of tallow and high rates prevailing for vegetable waxes.

If some 5 c.c. of ammonia be heated in a test tube and 3 decigrams of pure tallow stearin be added and shaken up, merely a faint turbidity is imparted to the liquid, but if the same treatment be applied to Japan wax an intensely white milky liniment is produced, and the same result is obtained with mixtures of Japan wax and tallow stearin.

When a little nitric acid, or aqua regia, is heated in a test tube and a little pure tallow stearin added thereto, no change of colour is observable, either during boiling or after re-cooling, but the stearin remains just as white as before. Vegetable waxes, however, when treated in this way, quickly turn yellow, and fumes of nitrogen oxide are evolved on heating. The same occurs in a lesser degree when mixtures of vegetable waxes and tallow stearin are employed. For the detection of paraffin, see p. 151.

THE REFINING, HARDENING AND BLEACHING OF TALLOW.

The tallow obtained from the crude fat by melting by one or the other methods mentioned is, even when carefully strained, not entirely free from admixed undissolved substances, *i.e.*, solid matters, and therefore cannot be used for all technical purposes. Though well adapted for the preparation of ordinary soaps, candles, etc., it is unsuitable for fine toilet soaps, and for this purpose requires to be refined.

The refining of tallow consists in melting the material, in presence of water, over a fire or by means of steam, with or without the addition of certain chemicals reputed to exert a purifying influence. The simplest and oldest method is by one or more re-meltings in an open pan over direct fire and with an addition of 5 per cent. of water; the steam melting process, wherein a current of steam is passed into the melted

fat, is new. It is an essential condition of the old clarifying process that the water should boil without ceasing and be thoroughly mixed with the fat by means of stirrers worked by hand or mechanical means, so that the latter substance is kept in a state of emulsion. When this mixing has continued for an hour the fire is drawn and the contents of the pan left at rest. During the slow cooling of the mass the lighter impurities rise to the surface and are skimmed off with a very fine strainer; the heavier and bulkier impurities, on the other hand, sink to the bottom along with the water, so that a mucilaginous layer of dirt and fat is formed between the strata of fat and water.

The precipitation can, naturally, only be complete provided the cooling of the tallow proceeds with extreme slowness. For this reason a very slow fire is kept under the pan, or the latter is covered up with a lid, cloths, etc., in order to retard the cooling as much as possible. Generally speaking, the fat in a pan of medium size will require about twelve hours to clarify thoroughly. The purified tallow is then removed by skimming or drawn off, through a tap at the side, and is placed in the transport packages, or, if a second clarification is desired, transferred to another pan. Very often common salt, alum, sal ammoniac, etc., are added to the water used for re-melting, an aqueous liquid of high density being thus obtained, from which the fat separates more readily than from ordinary water.

When steam is employed for clarifying it permeates the tallow, liquefies it and causes it to pass through a strainer of very fine mesh, which keeps back the impurities.

In addition to these two principal methods, others have been proposed with the object, partly of purifying, partly of hardening and bleaching, and also partly of sweetening the tallow by freeing it from unpleasant adherent smells. To effect the latter object (the cause of the smell being usually

rancid fatty acids the tallow is melted along with a solution of soda or borax, the *modus operandi* being otherwise exactly the same as when water alone is used.

Hardening the tallow is effected by various means. Apart from the old urine process (employed in slow-going soap-boiling establishments), the hardening process is based on a partial conversion of the olein in the tallow into solid elaidin by the action of acids, particularly nitrous acid. Alum, dilute sulphuric acid, potassium bichromate and manganese dioxide are also employed for hardening tallow, the products in most cases being oxy-fatty acids formed by the action of oxygen, whereby the final product is rendered harder and more brittle, and at the same time whiter in colour and almost inodorous.

According to one specification, a mixture of 500 grms. of concentrated sulphuric acid and 500 grms. of concentrated nitric acid is stirred into 100 kilos of melted tallow, and after allowing the acid mixture to react for a certain time is washed out again with pure water until the reaction is perfectly neutral, the tallow being finally heated over a gentle fire till all the water is evaporated.

When oxidising agents are employed, vapours are always evolved which contain whole groups of the volatile fatty acids: butyric, valeric, caproic and caprylic acids. Under the influence of nitrous acid, on the other hand, the reduction products of this acid appear in great quantity, and hydrocyanic acid is never absent. For this reason, hardening with this reagent can only be effected in closed vessels, the gases being suitably led away and rendered innocuous.

If the tallow obtained from crude tallow by melting at 60°-65° C. be allowed to crystallise at 35° C. and is then pressed, "prime press tallow" is obtained, whilst crude cuttings yield, under similar treatment, "press tallow seconds," the expressed liquid portion being in the former case classed as "prime margarin" and in the latter "margarin seconds".

The liquid portions of tallow can also be separated from the solids without pressure, since the solid crystalline substances have higher melting- and setting-points than the liquid products. To effect this separation the tallow is melted in large quantities (two to three tons at a time) by the aid of steam coils in large wooden vats, the vessels being then closed and left at rest for eight or ten days at a room temperature about 1° or 2° C. below the melting-point of the tallow. The fat has thus time to cool down very gradually, and the solid fatty glycerides crystallise in hard granules out of the liquid mass, settling on the walls and bottom of the vats in large conglomerate cauliflower-like aggregations. The liquid portion, the tallow oil, remains clear in the centre of the vessels and is then poured off, the crystalline lumps being left a while to drain and afterwards melted in water, poured out and allowed to set. In this way a product resembling press tallow is obtained. Nevertheless, neither this nor press tallow rightly deserves the name of "hardened tallow," since they are no longer tallow in its pristine state, *viz.*, stearin, palmitin and olein, but consist for the most part of stearin and palmitin, and therefore have an altogether different constitution.

In order to increase the whiteness of the product tallow is frequently *bleached*, the operation being oftentimes combined with the hardening process. Bleaching is effected exclusively with chemicals, *viz.*, chromic acid, manganese dioxide and hypochlorites. The various methods will be merely briefly mentioned in this place, having been already fully described in the author's works on *Vegetable Fats and Oils*.

1. *Bleaching with Chromic Acid or Potassium Bichromate.*

—100 kilos of the tallow to be bleached are melted along with 1 kilo (2·2 lb.) of sulphuric acid previously diluted with 6 litres (1·32 galls.) of water; thereupon 500 grms. (1·1 lb.) of powdered red chromate of potash are added and the whole gradually raised to boiling. When cold, the deposited acid

liquor is drawn off, and the tallow re-melted with water and washed in the warm until no acid reaction is detectable.

2. *Manganese Dioxide and Sulphuric Acid.* To 100 kilos of tallow is added 1 litre of concentrated sulphuric acid diluted with 30 litres of water, the fat being then melted and mixed with 1 kilo of the strongest manganese dioxide. The boiling mass is at first rendered black by the manganese compound, then bluish, and finally (when the latter is completely decomposed), white. It is then left to cool, the bleaching liquor drawn off, and the tallow freed from the chemicals by repeated meltings with water.

3. *With Hypochlorites.*—100 kilos of tallow are heated together with a solution of 1 kilo of soda in 10 litres of water, until the tallow is melted, a clear solution of 1 kilo of bleaching powder in 7 litres of water being stirred in and the mixture raised to boiling, whereupon sufficient dilute sulphuric acid to produce a faintly acid reaction is gradually and carefully incorporated therewith. After standing, the aqueous liquor is drawn off and the (melted) fat washed with water until no more acid can be detected.

Since, in the two last-named bleaching processes, noxious vapours producing coughing may, under certain circumstances, be given off, the operation must always be carried on with the bleaching vat placed under a metal cover discharging into the open air.

Uses of Tallow.—Medicinal: for ointments; cosmetics: for pomades; as a food-stuff: for cooking and frying, and in a special form as a substitute for butter; technical: for candles, soaps and lubricants of various kinds.

BONE FAT (KNOCHENFETT).

Raw Material.—Large hollow bones and short flat bones, or rather the spongy substance of the latter. Bones are filled with a soft yellow or reddish fat (bone marrow) which serves

to protect and support the blood vessels in the bone and also as formative substance for the white corpuscles of the blood. The bones worked up for their fat are those of the domesticated animals slaughtered for food and of the horse. As a rule, three products are obtained in the process: glue, fat and mineral matter, *i.e.*, phosphate of lime, the latter in the form of purified bone or as a manurial substance, or boneblack.

Preparation.—Originally the treatment of bones was confined to the extraction of the fat, the material being simply boiled in water and the fat skimmed off; in some cases also the glue, so far as the latter was dissolved by the boiling water, was recovered, the residual bones being worked up by the turner or bone worker, or ground down into bone meal.

The methods of manipulating bones employed at the present time may be directed to the attainment of four different objects:—

1. The recovery of fat and boneblack as the primary objects, with bone meal as a secondary consideration;
2. Fat and boneblack as the main products; glue and bone meal, by-products;
3. Fat, glue and bone manure or phosphate as the principal products;
4. The recovery of ammonia salts, in addition to the products named under 1 and 2.

In any case, whatever products are to be obtained, the separation of the fat is necessarily the first task to be performed. Even in the event that bone meal is the sole manufacturing product in view, the recovery of the fat is unconditionally desirable on account of its relatively high value, and the bones are of no greater value for manure with the fat left in than without it. In fact, they are the better for its removal, the fatty matter impregnating the bones retarding indefinitely their decomposition in the soil.

The form in which the bones should be used for recovering

the fat, the sole object at present in view, is always that of small lumps, such as are obtained by passing the bones through crushing rollers set closely together. In working with bones crushed by stamping, the meal and granules with which the unsorted lumps remain mixed unfavourably affect the progress of the operation, whether boiling or extraction, by clogging together and rendering the recovery of the fat more difficult.

Properly crushed bones contain no meal and but few granules, and are in this form best adapted for yielding up their content of fat.

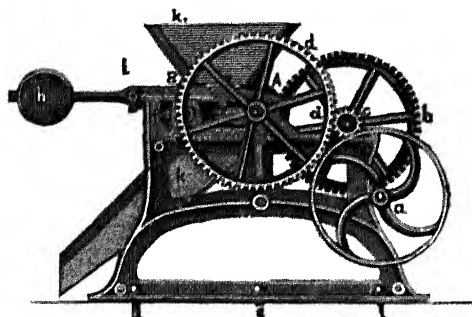


FIG. 65. Bone crusher. (Section.)

The recovery of fat may be effected in three ways :

1. By boiling,
2. By steaming,
3. By solvent extraction.

By simple boiling the bone cartilage is left almost entirely unaltered, the fat, however, being so imperfectly extracted by this operation that frequently only one-half the total quantity in the bones is recovered.

By steaming the bones a much greater yield of fat is obtained—over 90 per cent. being recovered by sufficing the operation ; at the same time, a large the cartilage is dissolved and converted into glue, the manufacture of this article does not fall within

the scope of the factory, is completely wasted, even forming a burdensome waste product, seeing that the glue liquor, unless quickly discharged into a large volume of running water, such as a river, very rapidly undergoes decomposition and pollutes the neighbourhood. The easiest method of getting rid of this glue liquor is by employing it for the irrigation of the land. Bone meal obtained from steamed bones is more easily dissociated than that prepared from unsteamed bones, on which account it has a more rapid fertilising action than the latter, and is, therefore, for this reason, usually considered by practical men as superior. Steamed bones when converted into

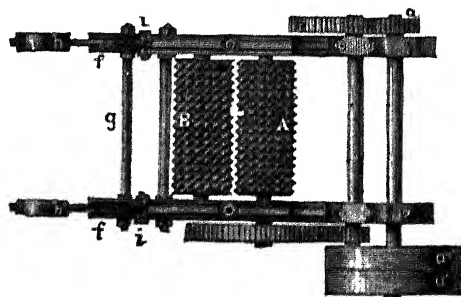


FIG. 66. — Bone crusher. (Viewed from above.)

boneblack are of lower value owing to the extraction of the gelatine; they yield a black of inferior decolorising power than unsteamed bones.

By the solvent extraction process the fat is almost entirely extracted, only a few parts per million at most being left behind in the bone. The cartilage, as well as the mineral matter, remains completely unaffected, and bones deprived of their fat in this manner may be used either for the preparation of a good bone meal, which will have an abiding manurial effect, or for the manufacture of boneblack. The solvent extraction method is recognised as producing the best results of any, and is coming largely to the fore.

1. *Bone Boiling.*—The oldest form of apparatus employed for bone boiling consisted of a large wrought-iron boiler set in brickwork and heated by direct fire. The pan was first filled with bones up to about 12 to 15 in. from the brim and water run in until the level of the liquid was about 8 in. below the edge, heat being then applied and continued until a fatty layer collected upon the surface, this being skimmed off by

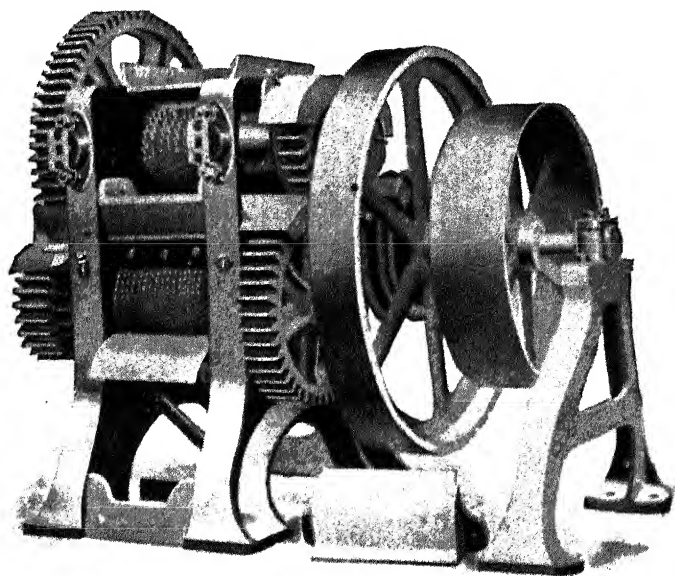


FIG. 67. Bone crushing machine (Rose, Downs & Thompson, Ltd.).

flat ladles as quickly as it formed and collected in a wooden vat, where it was left to set. Under the fatty stratum there was always found a certain quantity of dark-coloured glue liquor, which was poured off. As soon as only a scanty amount of fat was found to be rising to the surface of the water the operation was considered at an end, the glue liquor

was run off and the bones taken out of the pan to be then dried and subsequently worked up.

A slight improvement on this original practice of obtaining fat from bones consisted in placing the latter in a plate cylinder Fig. 60 perforated like a sieve in the lateral walls and suspended by a strong bow from a chain running over a pulley attached to a revolving crane, by the aid of which it could be lowered into the boiling pan.

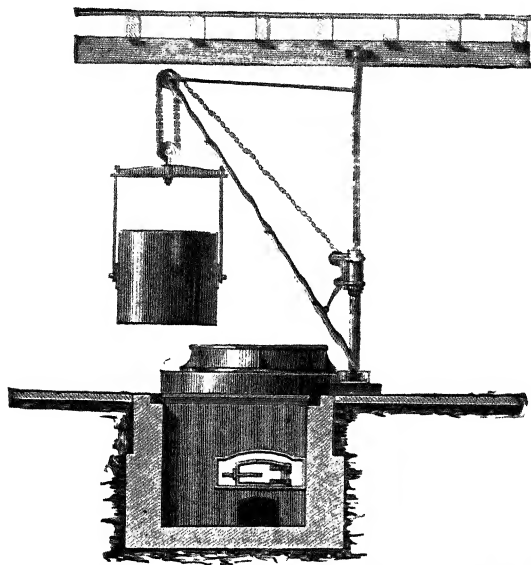


FIG. 68.—Bone-boiling pan with lifting gear and perforated vessel.

In this latter vessel the cylinder was allowed to remain until no further notable quantity of fat could be skimmed off the surface of the water, whereupon the cylinder was lifted out and replaced at once by another full of fresh bones, which were boiled in the same water, whereby a glue liquor was obtained of sufficient strength to repay for working up into glue.

With regard, however, to the suitability of the same water

for continuous use with fresh charges of bones, a difficulty soon arises, in that the viscosity of the liquor becomes so great that the ascent of the fat globules is hindered and they remain imprisoned in the liquor, water having to be added as a diluent to enable the fat to separate, besides which it tends to burn on the bottom of the pan.

Where steam is available, the boiling is effected by direct steam, which presents the advantage that large wooden vats may be used, the consumption of heat being relatively low.

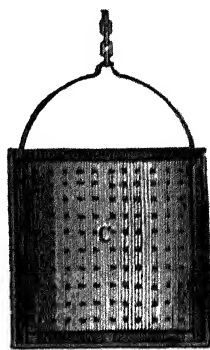


FIG. 69. Perforated vessel for holding the bones.

Direct steam may also be employed in the following manner: the previously mentioned perforated vessel being immersed in a wooden vat full of water, at the bottom of which is placed a coiled steam pipe, the steam from which heats the water to boiling.

2. *Steaming Bones.*—The temperature of steam increases according to the pressure to which it is subjected; it also forces its way more readily into the interior of substances than when in the liquid state (water). These two factors, conjoined with the circumstance that strongly heated fat is very fluid, it follows that bones subjected to the influence of fairly high-pressure steam yield a far larger quantity of fat than can be obtained therefrom by simple boiling.

Highly heated steam has, moreover, much greater solvent powers than hot water, and exerts this influence in the case of bones by converting a comparatively large amount of the cartilage into glue, so that in addition to obtaining a greater yield of fat from the bones by steaming, a more highly concentrated glue liquor is also produced than by mere boiling;

at the same time the steamed bones, being rendered more w the solution of an appreciable proportion of their

cartilaginous substance, are softened and can be more easily comminuted. The apparatus generally used—with slight modifications—for steaming bones takes the form displayed in Fig. 70. It consists of a cylinder 10 or 12 ft. in height, constructed like an ordinary boiler, of strong iron plates. In each of these cylinders, which are set up vertically and arranged in groups or "batteries," are two large apertures, E and A, which can be closed steam-tight by iron covers and bowscrews. In the upper convexity of the cylinder is fitted a pipe D communicating with the boiler, and on the opposite side of the pan is a short pipe H, both this and D being provided with cocks. At the bottom of the cylinder is a pipe L, also fitted with a cock and bent forwards, and the cylinder is fitted internally with a perforated false bottom S immediately over the domed bottom. Generally four to six, or in large works eight and more, steamers are united to form a battery, the discharge pipes L being led into a common outlet, whilst the steam pipes D are supplied from a main feed pipe. The batteries may be surrounded by brickwork, but it is preferable to mount them unenclosed and merely lagged with wood on a frame, the intermediate space between the boards and the steamer being packed with sawdust.

For filling the steamers quickly and with the smallest expenditure of power, it is advisable to set the crushing mill at such a height that the crushed bones fall direct into trucks which run on rails above the upper manholes of the steamers and are unloaded there by tipping. Another set of rails runs along in front of the second manholes A, so that the spent bones can be discharged direct into trucks and transported therein to the breakers.

As soon as a steamer is charged with bones it is hermetically closed, the tap H is opened and steam admitted to the bones by opening D. The steam first entering is condensed to water, being cooled by the bones, but after a short time

the temperature in the steamer will have so far risen to a point at which the steam no longer liquefies, when, after driving the air in the apparatus out through the pipe H it issues therefrom in a strong current. As soon as this is observed H is closed and the steam allowed to act on the bones, whereby the fat

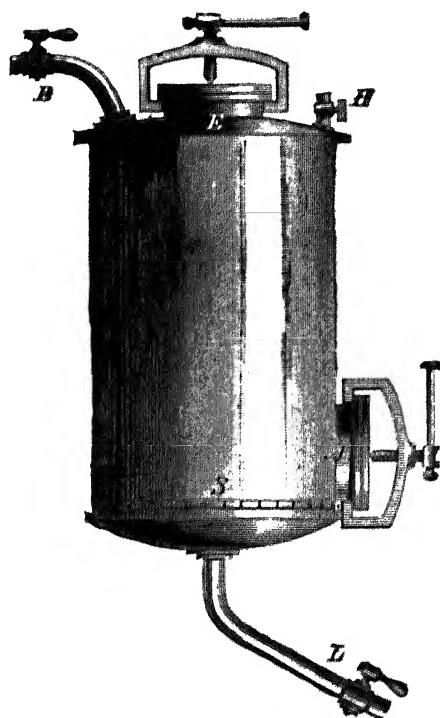


FIG. 70. — Bone steamer.

contained in them is melted and trickles down. At the bottom of the vessel there collects a liquid containing glue, rendered turbid by imprisoned fat globules, a thick layer of fat collecting on the surface. From time to time, at intervals of about an hour, the tap L is opened a little way whereby the pressure of the steam expels the glue liquor and fat with great force,

the tap being closed again when the characteristic sound indicates that steam alone is escaping.

Steaming, with periodical removal of the fat, is continued until a sample of the effluent liquid shows that no more fat is exuding. The liquid in the steamer is then expelled by the steam, the steam pipe D closed and the outlet A opened, whereupon the steam is turned on again forcing the greater part of the charge of bones out through A, an operation greatly facilitated when the perforated false bottom S is arranged to slope well forward. The liquids obtained from the steamer consist of melted fat and a fairly concentrated solution of glue, retaining a large proportion of fat. To separate these constituents the liquor is run into a steam-jacketed metal cylinder, the current of steam being regulated to produce sufficient heat to keep the glue liquor rather fluid, so that, on standing, all the contained globules of fat are enabled to rise to the surface. When the separation is complete the fat is run off through a tap at the side of the cylinder and the glue liquor immediately drawn off through a valve in the conical bottom of the vessel to the concentrating pans. The glue liquor being always fairly rich, it is advisable to work it up into glue in the factory, since, even if the resulting product be of somewhat low quality, the loss of valuable material sustained by the bone meal or boneblack is to some extent compensated for by its recovery. It is somewhat difficult, in the type of steamer described, to heat the mass quite uniformly, consequently the extraction of the fat occupies rather a long time, which results in a large amount of cartilaginous substance being converted into glue.

This may be remedied by an improved method of construction, wherein the steam pipe does not debouch into the top of the steamer but is modified in order to secure the better distribution of the steam through the charge of bones. The steam pipe (Fig. 71) is in this case extended to the centre of the steamer and then passes downward in the direction of the

axis almost to the bottom. A number of lateral apertures *a* are provided at various heights in the pipe, each of them being so protected by a small bent metal plate *d* that no solid products can pass into the tube, whilst, at the same time, the free discharge of steam is ensured. These protectors were found necessary to prevent the obstruction of the pipe with small pieces of bone.

The *modus operandi* of this arrangement is easy of explanation: The steam passes through the numerous apertures in the pipe uniformly heating the bones over a large area, the temperature consequently rising quickly to a point at which the fat melts out.

The entire aim of the process—recovery of the maximum amount of extractable fat in as short a time as possible and the consequent restriction to a minimum of the conversion of cartilage into glue—is attained by this very simple arrangement. Finally, by this method of construction the diameter of the steaming cylinders can be increased and the bones still heated uniformly.

Bone fat is valued according to the "titer" in the same way as tallow and also according to its freedom from impurities. The following analyses of bone fat are by F. Jean:—

Sample.	Moisture.	Impurities.	Gelatine.	Titre Test.	Free Fatty Acids.
	Per Cent.	Per Cent.		" C.	
1	0.80	—	—	30.8	—
2	0.90	0.1	—	42.6	6.53
3	1.50	trace	—	39.4	3.61
4	2.00	0.12	—	42.3	—
5	0.67	0.02	—	44.5	—
6	4.42	4.87	0.664	36.9	—
7	7.36	0.60	1.73	36.8	—
8	6.53	2.03	1.20	38.3	—
9	0.96	4.92	—	39.8	—

vent Extraction.—The solvent extraction process is, stated, the only one by which it is feasible to ob-

tain from bones the contained fat in its entirety and at the same time preserve the cartilaginous substance perfectly unaltered, so that the best quality of boneblack may be prepared from the residue. Hence there is no need to undertake the manufacture of glue in order to compensate for the loss of valuable constituents—a consideration of great value in simplifying the work of the establishment.

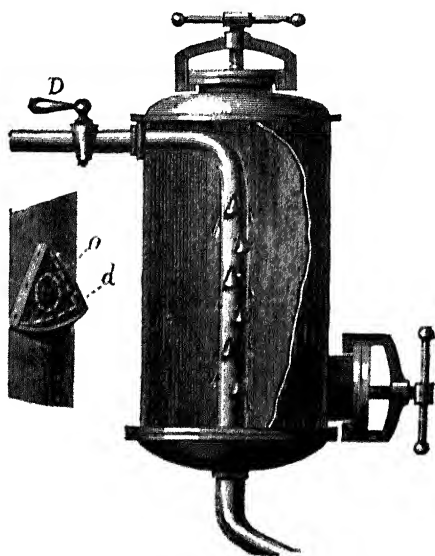


FIG. 71.—Friedberg's improved bone steamer.

The recovery of fat from bones by this process is based on the property of certain liquids, such as ether, carbon bisulphide, petroleum spirit, benzol, etc., of extracting fat, especially when warm, bringing it into solution and then yielding it up again as a residue on simple distillation. In most cases use is made of the cheap petroleum spirit (benzine) available in large quantities, a special apparatus being employed on account of the great care necessary in view of the explosive and inflammable nature of the solvent. Among the apparatus

for extracting fats, those of Deiss, Vohl, Seiffert, Linchner & Merz and Wegelin Hubner are best known, all of which are described in the author's work on *Vegetable Fats and Oils*. See also pp. 66 to 77 of this book. The extraction of the comminuted bones is effected in precisely the same manner as there indicated for oil seeds and oil fruits.

Properties.—The bone fat obtained by merely boiling the bones is, except when perfectly fresh bones are used (and this is very rarely the case), of very inferior quality, dark yellow to light brown in colour and of repellent smell. The colour is due to the dark coloured decomposition products of the putrefying bone cartilage, the evil smell arising from similar products, fats in general being characterised by the affinity they exhibit for absorbing and retaining odoriferous substances.

Bone fat prepared by the steaming process differs little from the last-named class with regard to colour, but as regards smell it is infinitely superior. The same partly decomposed bones that, in the ordinary method of simple boiling, would yield a fat with a repulsive odour, will, if steamed, give a much less unpleasant-smelling product, the difference being attributable to the higher temperature employed, whereby the greater part of the malodorous substances is vaporised and carried off with the steam; besides, there is less local heating and therefore less chance of destructive decomposition.

The external appearance of the fat derived from bones varies considerably, that from perfectly fresh bones being pale yellow in colour and free from smell, whilst that from old bones is dark yellow to brown; the consistency is either quite fluid, semi-fluid or like lard.

The constants of bone fat are as follows:—

Specific gravity at 15.5° C.	0.914-0.916
Solidifying point	15°-17°
Melting-point	21°-22°
Free fatty acids	14.8-26.5
Saponification number	190.9-195
Iodine number	46.3-55.8
Holmer number	86.1-94.1
Ash.	0.11-2.40

Uses of Bone Fat.—Solely technical; in the soap and lubricating oil industries.

Refining Bone Fat.—For the purpose of utilizing bone fat it is essentially necessary to subject it to a process of purification in order to destroy the odours and colouring matters contained in it; this process is carried out with either nitric acid or potassium bichromate, both of which exert a powerful oxidising action. In refining with nitric acid 1 to 2 parts by weight of acid are taken per 1000 parts of fat, the exact amount depending directly on the degree of impurity.

Wooden vats, containing wooden appliances for stirring, and fitted with a steam pipe at the bottom, are used. The fat to be purified is placed in the vat, where it is melted by steam and heated to 70°-80° C., the nitric acid being then run (from a glass vessel fitted with a glass tap) in a thin stream into the fat, during which time the stirrers are kept in continual motion. At intervals samples are withdrawn from the vat, cooled rapidly to cause them to set, and examined to obtain an idea of the condition of the fat. If the purification appears to be sufficient the stirrers are stopped and the fat washed, with which object it is allowed to run out in a thin stream into a second vat placed at a lower level and more than half full of water, besides being fitted with a steam pipe and set of stirrers. By admitting steam and simultaneously working the stirrers an intimate admixture of the fat and water is effected; after a short time the whole is left at rest, the water, after separation, being finally drawn off and replaced by a

fresh supply, whilst the fat is washed again in the same manner and is then considered sufficiently purified.

By replacing the nitric acid by a mixture of equal parts of nitric acid and sulphuric acids, the purifying process is considerably accelerated, but the loss is greater than when nitric acid alone is used.

The second purifying process is that in which sulphuric acid and potassium bichromate are used. One part by weight of bichromate is dissolved in a very little water, and the solution placed in a stoneware vessel, 2 parts by weight of sulphuric acid being poured in and stirred with a glass rod, the vessel being then left, covered up, for a couple of days. When sulphuric acid and potassium bichromate are brought into contact, chromic acid is liberated which dissolves in water to a red solution, whilst the potassium bisulphate formed at the same time crystallises out at the bottom of the vessel. From $\frac{1}{2}$ to 2 per cent. of this chromic acid solution is added to the fat, according to the degree of impurity of the latter, and the stirrers are set in motion.

When the reaction is concluded the fat is washed, at first with but a small quantity of water, which becomes tinged green or violet by the resulting chromic compounds, and these washings from several operations are sometimes collected and sold to colour manufacturers.

By using a sufficiency of bichromate, even a very impure bone fat can be rendered perfectly white and free from smell, and this class of product sells at such a price that it well repays the high cost of refining.

Krätzer employs for bleaching bone fat a mixture of 500 parts by weight of the fat, warmed to 70-75° C., and 5 parts of 30° soda lye containing 2.5 parts of common salt, leaving the whole to stand for six to eight hours or overnight. The clear settled fat is then cooled to 40° C., and to it are added successively a solution of 2.5 parts by weight of potassium

chromate and 7.5-10 parts of 22° fuming hydrochloric acid, fixed in by means of the stirrers, which are kept vigorously moving until the oil finally exhibits merely a green tinge with white froth, a sign that it is properly bleached. When this result is arrived at, some 20 gallons or so of water at 75° C. is sprinkled over the surface from a watering-can and the hole covered up and left to stand, steam, if available, being blown in for ten to fifteen minutes.

By this method of washing with hot water or steam the acid remaining in the bone fat is washed out and carried down.

A bone fat bleached in this manner is, at the ordinary temperature, soft and lard-like in appearance, pure white or faint yellow in colour, and is specially characterised by the slowness with which it becomes rancid when exposed to the air.

BONE OIL (KNOCHENÖL).

In order to obtain the bone oil from bone fat, which is done by precipitating the more readily congealable stearin group of fats, the fat is exposed to a low temperature (about 2°-9° C.), whereby the solid fat separates out and the oil can then be poured off. According to the Polytechnic Society of Berlin, much better results can be obtained by dissolving the fat in benzine and exposing the solution to cold. The clear liquid is poured away from the solid fat, and when freed from the solvent by distillation leaves pure bone oil.

NEATSFOOT OIL (KLAMENÖL).

Raw Material.—The hoofs of cloven-footed animals: oxen, sheep and goats.

Preparation.—The feet of the ox and the sheep contain a very liquid and stable fat. In order to obtain this substance the fresh feet are laid in cold water to wash away the adherent blood and, after the sinews have been removed, are

then placed in sufficient boiling water to cover them. After immersion for a quarter of an hour therein they are taken out, the claws cut away and the feet split to separate the toes from the larger bones, the latter being then boiled in water by the aid of steam, whilst the former, which yield up their fat less readily, are placed in a pan and boiled over direct fire in the water that has already served for boiling

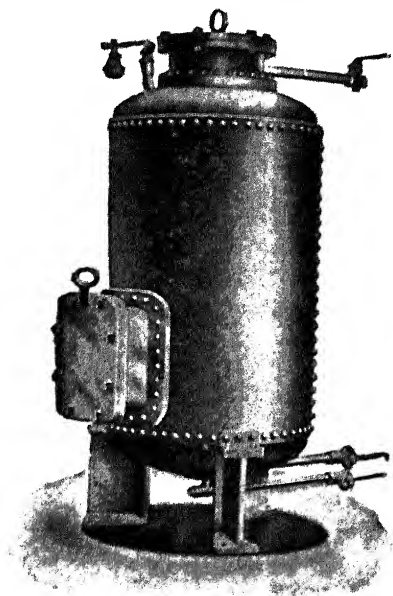


FIG. 72.—High-pressure bone digester (Rose, Downs & Thompson, Ltd.).

the large bones. After a prolonged boiling the liquid is allowed to clarify, whereupon the fat rises to the surface and is poured off. The oil obtained in this manner deposits after some time a dirty semi-fluid fat, from which the liquid portion is separated by decantation. This oil is pale yellow in colour and thin at ordinary temperatures, setting only at a few degrees below zero C. It contains much olein and but little stearin, does not easily turn rancid nor does it readily

thicken. After the fat still remaining in solution has been as far as possible separated by crystallisation and filtration, the liquid product forms a very good lubricating oil for clocks and delicate machinery.

According to Th. Chateau, the preparation of neatsfoot oil is carried out in the following manner: The feet ("trotters") of about 100 sheep are treated for twenty minutes in a

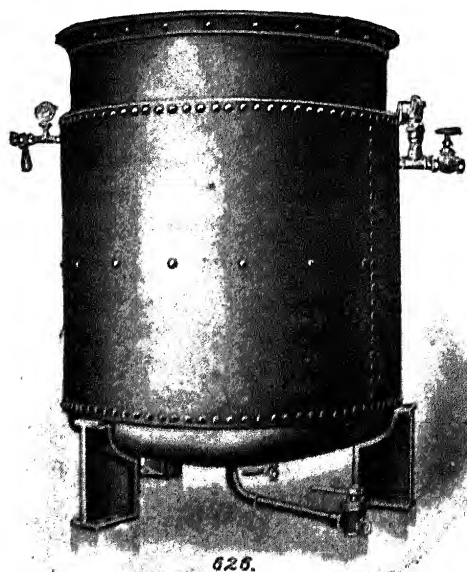


FIG. 73. Steam-jacketed bone digester (Rose, Downs & Thompson, Ltd.).

pan of water warmed by the aid of steam to 75° or 80° C. When the woolly hair comes away easily the pan is emptied, the feet scraped and the hoofs removed. The feet thus cleaned are tied up with string into bundles of eighteen and then subjected to extraction by boiling in water until the greater part of the contained oil is recovered, whilst the feet themselves are sold in the half-cooked state. From 100-125 bundles of eighteen are treated at a time. The yield of fat

is somewhat variable, ranging from 1 lb. to 21 lb. per 100 sheep. The feet of animals that have had to traverse long distances before being slaughtered, such as is the case with American sheep, yield merely traces of neatsfoot oil. The boiled feet are immediately laid in a stream of cold water, and when cooled are offered for sale. The oil obtained in this manner has a specific gravity of 0.915, is translucent and grey in appearance, but clarifies on standing or by several filtrations, and is then very pale yellow in colour. Chateau, in his communication, reports that the majority of commercial neatsfoot oils examined by him were composed of other fats.

E. Geissler points out that neatsfoot oil is, on account of the method of preparation, very often rancid, and that he has examined oils of this class containing 10-15 per cent. of free fatty acids.

According to Schaedler, true neatsfoot oil from the hoofs of oxen is mostly mixed with that from the feet of the sheep.

Benedikt distinguishes between ox neatsfoot oil, sheep's-foot oil and horse-foot oil, and states that commercial neatsfoot oil is generally a mixture of oils from the hoofs of oxen, sheep, horses and swine. Data for the accurate differentiation of these oils are still entirely lacking.

Properties.—Neatsfoot oils are straw yellow, inodorous, of agreeable flavour and generally set only below 0° C.

Constants of neatsfoot oil :—

Specific gravity at 15° C.	0.9152-0.9165
Solidifying point	- 3°-4°
Saponification number	194.3-199
Iodine number	67.1-76
Refractive index at 20° C.	1.4681

Adulterations.—Purified neatsfoot oil is in good demand as a lubricant ; it is often adulterated with refined cotton-seed oil, rape oil and mineral oils, the detection of which is not difficult. Any admixture of pale fish oils can be recognised

by the smell evolved when rubbed on the hands ^{otherwise} by phosphoric acid.

Uses.—Technical: as a lubricant for delicate machinery, clocks and so forth.

HORSE FAT, HORSE GREASE (PFERDEKAMMFETT, PFERDEFETT).

Raw Material.—Chiefly the fat found in the neck (under the mane) of the horse, and in addition all the fatty portions of the carcase.

Preparation.—The fat from the leaf, back, neck, and kidneys after separation from its integument is collected and melted—by steam if on a large scale, or simply by hot or boiling water in small works—then strained and placed to set in suitable vessels.

Properties.—The actual mane fat of the horse is pure white and inodorous, softer in consistency than lard, melts at 30° C. and consists of 70 parts of olein and 30 parts of a mixture of stearin and palmitin.

The other fat from this animal is thick, almost like pomade, and separates on standing into a solid portion and a supernatant liquid. At 10° C. it resembles lard, and at 8° C. tallow; it is of a dirty yellow-brown colour and possesses a characteristic fatty odour. The fat has slight drying properties and Farnsteiner was successful in preparing from it a linolic tetrabromide in amount equal to about 10 per cent. of linolic acid.

The marrow fat obtained from the large hollow bones of the horse is a waxy-yellow, greasy fat, becoming hard in the air; it begins to run at 65° C. and is at 86° C. thick like syrup. By the aid of soda a very solid white soap can be prepared therefrom.

The constants of horse oil are as follows :—

Specific gravity at 15° C.	0.916-0.933
Melting-point	34°-53°
Acid number	1.73-2.44
Saponification number	195.1-196.8
Hehner number	94.5-95.5
Reichert-Meißl number	1.64-2.14
Iodine number	71.3-86.3
Refractive index at 25° C.	1.4667-1.4697.

Uses.—Medicinal : for ointments in veterinary medicine ; cosmetic : for pomades intended to promote the growth of the hair. For softening leather, lubricating machinery, greasing wool, making soft soaps, for which it seems specially adapted. It has also been used for adulterating lard.

ANIMAL OIL (THIERÖL): DIPPÉL'S OIL.

Raw Material.—The tar resulting from the dry distillation of bones.

Preparation.—The crude bone tar is placed in a still connected with a warm condenser. Liquid distils over rather quickly at first, and when the distillation shows signs of slackening the fire is made up stronger, so that, finally, nothing remains in the still but a porous black residue consisting of carbon which is utilised for fuel. The distillate is pale to dark yellow in colour, of oily appearance, and characterised by the fluorescence it exhibits, the surface of the oil, when viewed at a certain angle, displaying a coloration inclining to blue or red. It should be mentioned that when a parcel of bone tar has been once distilled, it must be worked up completely without cessation, since otherwise the oil will turn brown by oxidation during storage.

The distillate is shaken up with strong hydrochloric acid, arated therefrom, washed with water and rectified in

By this means a series of products which readily and give rise to dark colorations are removed ; in

this way it is possible to prepare an oil that will remain light coloured.

Since the action of oxygen is increased by high temperatures, an arrangement has been devised for re-distillation, after the acid treatment, in a current of carbonic acid (carbon dioxide), in which case a copper vessel may be used for rectifying. The current of carbonic acid gas must, however, be strong enough to fill the receiver in which the distillate is collected, in order to prevent the animal oil coming in contact with the air. By adhering to these precautions the distillate from the crude oil may be made to yield after two rectifications a product which is almost colourless, and will remain fairly so if air be excluded. On exposure to the air it becomes dark-coloured, even though several times rectified, to prevent which discoloration, as far as possible, the oil should be stored, after rectifying, in air-tight bottles.

Properties.—Thin flowing and pale yellow when fresh, but brownish-black from oxidation when aged, with repellent smell. On treatment with sulphuric acid it is rapidly converted into brown clotted masses.

Uses.—Medicinal: as a popular curative agent; technical: in the wool-dyeing industry.

CHAPTER V.

SEA-ANIMAL AND FISH OILS (THRANP).

THE fish oils include those liquid animal fats that are obtained from the fatty accumulations in the bodies of the large marine mammals; from the liver of various smaller or larger fish; and finally by pressing certain species of fish, all of which inhabit salt water.

All fish oils are, at the ordinary temperature, more or less fluid, pale yellow to dark blackish-brown in colour, and mostly endowed with a suffocating, more or less disagreeable smell and taste. Of the fish oils—whose chemical constitution is still insufficiently known—one group is classed with the liquid waxes (sperm oil and the spermaceti prepared therefrom), and they are all very easily distinguished, by their behaviour on saponification, from the remaining oils and fats.

Sperm oil and spermaceti are devoid of glycerides, but contain instead ethers of the higher fatty alcohols; and shark oil and all the marine animal oils with a specific gravity below 0.880 at 15° C. may be classified in the same group, since the great majority of the glycerides have a higher specific gravity than 0.914. Little is known respecting the fatty acids occurring in the form of tri-glycerides in fish oils. The place of the oleic acid found in other oils seems to be occupied here by physetoleic acid, but it follows, from the very high iodine number of fish oils, that large quantities of a glyceride of an acid poorer in hydrogen are present; this cannot, however, be linolic acid, because fish oils are not endowed with drying properties.

Most of the fish oils are blackened by gaseous chlorine, whereas all other fats and oils are bleached by this reagent. The true, and to some extent the wax-like, fish oils as well give highly characteristic colour reactions: caustic soda of specific gravity 1.34 and syrupy phosphoric acid producing a red coloration; nitric acid, sulphuric acid and nitrosulphuric acid giving black or violet-black colorations.

A large number of fish oils are met with in commerce and are classified according to whether they are derived from the carcase fat (blubber), the fatty liver or the whole fish, and also according to the genus of the animal yielding them. They are all employed either for medicinal (cod-liver oil) or technical purposes.

By the term fish—or train—tallow (*Thrantalg*) are indicated the solid fats (stearins) separating out from fish oils at temperatures near freezing-point, or obtained by pressure at such temperatures. Crude whale oils yield "whale fat," whilst the liquid portions are known as "expressed whale oil" (chiefly consisting of physetolein).

The fish oils met with in commerce may be classified as follows:—

1. *Whale oils* (train or blubber oils: whale, sperm whale, fin-back whale, Arctic sperm, dolphin, porpoise, walrus, round-headed dolphin oil.

2. *Seal oils*: Archangel, Greenland, Newfoundland, South Sea, Caspian seal oil, walrus and Swedish "Three crown" oil.

3. *Fish oils*: herring, sprat, pilchard, sardine, sardella, menhaden oil, Swedish, Russian, Italian, and Spanish fish oil.

4. *Liver oils*: cod-liver oil, coal-fish oil, merlangus oil, pollack oil, sea-pike oil, shark's-liver oil, ray oil, ray-liver oil, Japan fish oil.

The fish oils obtained from the blubber of the entire fish, or portions thereof, by boiling, contain in their natural state more or less animal gelatine (glue) derived from cartilaginous

matter. The various kinds of fish oil have different odours and flavours, which are indescribable, but when experienced a few times, fix themselves in the memory and form the taste especially) a sure, if not agreeable, means of recognition.

The various kinds of fish oils—pure seal oil, whale oil, liver oil, train oil—can be distinguished by the aid of fuming nitric acid and sulphuric acid, the colour reactions obtained being nevertheless unreliable.

The violet colorations given by liver oils with sulphuric acid do not result from the presence of biliary colouring matters, but, according to Salkowsky, cholesterol, the colouring matter (lipochrome) discovered by Kühne, and the fatty acids themselves take part therein. Further assistance in differentiation, and perhaps the detection of adulteration, should be afforded by the difference of solubility in hot alcohol, in that the latter will take up 4 per cent. of fish oil, 7 per cent. of liver oil, 15 per cent. of seal oil and its own volume of whale (blubber) oil.

The fish oils are, for the most, only adulterated with lower grade train oils and rosin oil, the medicinal oils rarely with vegetable oils (such as sesame and cotton-seed oil), which latter, by the way, can be detected by the claidin reaction, fish oils remaining clear and transparent whilst the last-named oils deposit claidin after some time, the fatty layer being thereby rendered thick and opaque.

Further particulars are given under the different headings relating to fish oils (see table on opposite page).

I. TRAIN OR BLUBBER OILS.

Raw Material.—The fat of marine mammalia—sea cow, walrus, manatee, dolphin, porpoise, sperm whale, Arctic sperm whale, Greenland whale.

Preparation.—The dead carcasses are hauled on deck or, on land, on shore, and the cutting out of the blubber

is at once begun. The men stand on small platform, let down the side of the vessel, and cut strips, about a yard wide, in the blubber (which is 18 to 30 inches thick) right round the carcase, over the back and belly; then attach a rope to the strip and haul at the windlass, whereby the rope is tightened and the blubber draws loose, the men cutting it away from the carcase with sharp spades, so that the whole envelope of blubber is removed spirally by turning the body over, and is hauled on deck.

COLOUR REACTIONS OF THE FISH OILS WITH ACIDS.

	Formic Acid. A. 1. Dilute (1:10).	Sulphuric Acid. Dilute (1:10).	Nitric Acid. Dilute.
Seal oil.	Red-brown.	1. Yellow, then reddish brown, finally brown and almost black colour.	Yellow, then reddish.
Whale oil.	Brownish, then brown, finally blackish brown.	Brown, then blackish brown.	Yellow, then reddish, later a dirty brown.
Cod liver oil.	Blood red, then brownish red to brown.	Violet to blackish violet.	Yellow red, then brick red, finally red brown with violet tinge.
Fish oil.	Brown.	At first greenish, then brown, finally quite black.	Yellow, then greenish, under strongly brown.

The blubber is first placed 'tween decks, and is there cut up by machinery into smaller pieces, which are packed in casks, to be subsequently stowed in the hold. In many

instances, however, it is immediately melted down on deck, in an iron pan set in brickwork, the oil being strained and filled into casks, whilst the residue (greaves) serves as fuel.

The blubber in the casks begins, after prolonged storage, to putrefy in consequence of the decomposition of the tissues and other animal matters, and a part of the oil runs out spontaneously and is collected in special recipients. When the vessel arrives at her destination the blubber still left in the barrels is melted down over a fire or by steam, and the self-run oil is also heated to about 100° C. to allow the impurities to subside. In some places the blubber, after being cut up, is melted in large pans 14 to 20 feet high, by direct steam, for five to eight hours. The carcase itself, when stripped of blubber, is either thrown back into the sea or treated for the preparation of manure.

The dorsal fat yields darker oil than the belly fat, and is treated separately. The yield ranges from 6 to 20 tons of blubber per whale.

When the work is performed in a rational manner, considerable quantities of oil, though of inferior quality, are obtained by treating the flesh and bone in the carcase with high-pressure steam, the residual portions being dried and sold as manure.

Smaller marine mammals, such as the porpoise, are disembowelled, cut into pieces, and the flesh and fat, as well as the bony skeleton, extracted by boiling over direct fire or by steam.

(a) *Porpoise Oil*.—Extracted from the porpoise (*Delphinus phocaena*). The oil from the "jaw" differs from the oil from the "blubber," is pale yellow, brown-yellow or brown, with a smell resembling that of sardella oil, but loses this on exposure to air, and assumes a deeper shade of colour. The oil is neutral to litmus paper, but absorbs oxygen from the air, and then has an acid reaction. Its specific gravity

at 15° C. = 0.9258; setting-point. - 15° C. Cold alcohol dissolves 1.2 per cent.; boiling alcohol, 20 per cent. Reichert number, 23.41 c.c. KOH; Saponification number, 195-250.61. Iodine number, 88.3-119.4. The constituents of this oil are: glycerides of physetoleic, oleic, stearic, palmitic and valeric acids.

(b) *Dolphin Oil*.—From the dolphin or black fish (*Delphinus globiceps*). *Round-headed dolphin oil* is pale yellow in colour and has a strong fishy smell; specific gravity at 15° C. = 0.9266; at 20° C. = 0.9175; Reichert number, 1.6 c.c.; saponification number, 197.3-203.4; iodine number, 99.5-126.9. One hundred parts of boiling alcohol of specific gravity 0.812 dissolve 40 parts of the oil, the solution becoming turbid at 50° C.; 100 parts of boiling alcohol of specific gravity 0.795 dissolve 60 parts. When exposed to low temperatures the oil at 5° C. to - 3° C. deposits spermaceti, and when thus freed from the latter substance dissolves in equal proportions in boiling alcohol, the solution exhibiting a weak acid reaction, which disappears on the addition of water.

The oil obtained from the "jaw" differs markedly from the oil from the "blubber," containing more volatile fatty acids, the Reichert number being 66, the Hehner number also 66, the saponification number 290.0, and iodine number 32.8. The "jaw oil" is a fine product.

(c) *Narwhal Oil*.—Very pale in colour; almost white.

(d) *Sperm Oil*.—*Southern or cachalot oil*, from the blubber and head of the southern sperm whale (*Physeter macrocephalus*), of which a large fish will yield from 70 to 90 tons and 50 cwt. of spermaceti, is pale yellow to slightly brownish-yellow, clear, with a decidedly fishy smell and fairly fluid, with a specific gravity of 0.880 at 15° C. At 6° C. it begins to deposit spermaceti, and stearin at 8° C. Two vols. of the oil are soluble in 10 vols. of alcohol at the ordinary temperature; and 7 vols. in 10 of boiling alcohol. It is miscible

with ether in all proportions, and sets slightly under the claidin test, thus differing from all other fish oils.

The spermaceti is separated from the oil by filtration and used in making candles. The alimentary canal and the bladder of this whale contain the highly-prized perfume known as ambergris.

(e) *Arctic Sperm or Bottlenose Whale Oil*.—This product is colourless to brown, of slightly repellent smell, thin, with a specific gravity of 0.885 at 15° C.; absorbs oxygen from the air and thickens, its specific gravity at the same time increasing. It contains a relatively low proportion of solid fatty acids since it remains liquid below 5° C., with merely a slight turbidity, and only below -2° C. becomes gruelly in consistency. It dissolves in 25 parts of cold and 2½ parts of boiling alcohol, but separates out again, for the most part, on re-cooling.

When treated with nitrous acid the oil exhibits a tendency to set. It contains only about half as much oxygen as other fish oils, and consists of:—

79.87	per cent.	carbon.
13.36	"	hydrogen
6.77	"	oxygen
100.00	"	

and in addition to physetoleic acid, etc., about 1 per cent. of spermaceti. Doeglic acid, $C_{19}H_{36}O$, is probably a mixture of physetoleic acid and an allied acid, this being apparent from the uneven number of carbon atoms in the formula.

There is not much difference between the two kinds of sperm oil, as shown by the following table of constants:—

	Cachalot Sperm Oil.	Arctic Sperm Oil.
Specific gravity at 15° C.	0.880	0.880
" " " 100° C.	0.833	0.827
Saponification number	133.4-147.1	126.4-135.0
Iodine number	70.93	80
Reichert number	1.4	1.4
Fatty acids per cent.	60.64	61.65
Alcohols per cent.	39.41	37.44
Mannene test	51° C.	42° C.
Refractive index	1.4636	1.4655

These oils differ from all other oils in consisting not of glycerides essentially but of ethers, compounds of alcoholic bodies with the fatty acids.

Sperm Oil.—Owing to the trouble experienced in the use of sperm oil for burning in safety lamps of the Davy type, A. G. Blakely and E. A. Reilly have made a careful investigation of a number of samples of sperm oil, some of which burnt well and others badly; the observations including the determination of the usual chemical and physical constants, and, in addition, certain physical characteristics, such as "flake test," "cloud test," and "pour test," which are variations of the solidifying point and viscosity tests, and appear to be new. Further, they obtained the fatty acids and the alcohols separately, and also applied to them the usual methods of analysis, so that the investigation represented a considerable amount of work. The samples which gave trouble, *i.e.*, produced an incrustation on the wick, were somewhat abnormal in composition, and probably adulterated. The composition of the samples of pure sperm oil varied beyond the limits usually quoted as given below, compared with the two samples which gave most trouble:—

	Pure Sperm Oil.	Badly Burning Samples
Specific gravity at 15.6° C.	0.876-0.883	0.881-0.884
Refractive index at 15.6° C.	1.4643-1.4669	1.4893-1.4900
Saponification value	127.7-142.2	142.7-145.7
Acidity calc. as oleic per cent.	0.04-0.60	1.17-1.42
Iodine value (Hanus)	83.1-89.6	62.4-64.7

	Pure Sperm Oil.	Badly Burning Sample.
Fatty anhydrides per cent.	52.4637	59.9601
Alcohols per cent.	33.6462	36.4278
Flake test °F.	37-52	—
Cloud test °F.	34-46	—
Pour test °F.	24-38	37-37
Flash point °C.	245-265	230-270
Fire point °C.	285-300	285-300
Viscosity (Tagliabue) at 70° F.	106-116	111
" " " 212° F.	88-97	86
" (Saybolt) " 100° F.	110-127	—
" " " 130° F.	74-88	—
" " " 210° F.	46-50	—
Fatty Acids—		
Specific gravity at 15.6° C.893-.899	.895-.895
Refractive index at 15.6° C.	1.4580-1.4623	—
Neutralization value	186.8-216.2	222.8-224.8
Iodine value (Hanus)	82.2-91.8	64.9-66.2
Titre test °C.	6.2-13.6	12.0-12.0
Alcohols—		
Refractive index at 60° C.	1.4383-1.4495	—
Iodine value (Hanus)	63.9-72.5	39.3-41.4
Melting-point °C.	20.0-27.0	34.0-35.0
Saponification value of acetate	189.5-216.0	198.3

Experiments were made as to the effect of free fatty acids on the burning properties of the oil, using varying proportions of oleic acid. Very little influence was found with quantities under 4 per cent., only a slight crust being formed; but with 5 per cent. or upwards a thick crust was formed, and the oil therefore became quite unsuitable for burning purposes.

SPERMACETI (WALRATH, SPERMACEET).

Raw Material.—The fat found in the skull cavity of the cachalot or sperm whale, and in other species of dolphin. In the living animal the spermaceti occurs dissolved in sperm oil, but separates out after death.

Preparation.—When the skull is opened the fatty mass is collected and stored in large vessels, the liquid sperm oil being filtered off and the residue, consisting of a mixture of

spermaceti² and sperm oil, is pressed, leaving behind a crystalline fatty mass. This is next treated with a weak lye of caustic potash or soda, to saponify and remove any adherent oil, after which it is repeatedly washed with water and remelted over boiling water.

Crude spermaceti, which is seldom met with in commerce, forms plates with a foliaceous structure and of about the thickness of the finger; is transparent and has a fishy smell. The purified article comes to market in the shape of semi-transparent white, broad, foliaceous, crystalline lumps with a feel like talc. According to Benedikt spermaceti is obtained from sperm blubber by pressing.

Properties.—Spermaceti is solid, tasteless, inodorous, and may be crumbled between the fingers.

Specific gravity at 15° C.	0.905-0.960
" " 60° C.	0.8358
Solidifying point	41°-49°
Melting-point	41°-46°
Acid number	0.09-1.35
Saponification number	120.6-134.6
Iodine number	3.52-9.3
Fatty acids, per cent.	49.78-53.45
Alcohols	" 	49.54.27

If the product be treated several times with hot alcohol, the residual substance has a melting-point about 5° C. higher than before. It may be distilled at 360° C. without any noticeable decomposition. When applied in a melted condition to paper, it leaves no grease spot behind. It is very slightly soluble in cold 98 per cent. alcohol, and quite insoluble in 90 per cent. spirit, but, on the other hand, dissolves easily in hot alcohol, and crystallises out again for the most part on cooling. Its solutions do not redden litmus paper. According to Schaedler, 1 part of spermaceti is soluble in 40 parts of boiling alcohol of specific gravity 0.831, the greater part separating out on cooling. It is, moreover, but slightly

soluble in benzine and petroleum spirit, though readily soluble in ether, chloroform, or carbon bisulphide. Spermaceti is a mixture of various fats, but contains no glycerides; on re-crystallisation from alcohol the crystalline substance fat—cetin—cetyl palmitate, $C_{18}H_{38}.O.CO.C_{17}H_{35}$, formerly called cetyloxyde cetate or athal athalate, separates out. According to Benedikt, spermaceti can be easily saponified with alcoholic caustic potash, cetyl alcohol being precipitated on diluting the solution with water, but, according to Schaedler, spermaceti can be saponified only with difficulty. The elementary composition of spermaceti is as under:—

Carbon	80.03 per cent.
Hydrogen	13.25 "
Oxygen	6.72 "

Adulterations.—Spermaceti cannot be easily falsified, since its properties are very remarkably modified by any admixture that may be made, and adulterations are readily recognisable by the increased hardness of the substance, its lack of nacreous lustre, and the small foliaceous crystalline structure. According to Schaedler, wax is frequently added to spermaceti, but the quantity cannot be large, otherwise it would raise the specific gravity and melting-point or cause the ethereal solution to appear milky. Tallow betrays itself by its odour when melted or by the smell of acrolein produced when a lighted wick steeped in the substance is blown out; moreover, spermaceti containing tallow leaves a greasy mark on paper. If stearic acid be added, the spermaceti is hardened, the crystals are reduced in size and the mass foams when heated with soda solution. The saponification value serves for the detection of paraffin.

In order to test spermaceti for stearic acid, a sample is melted in a basin, ammonia added, and the whole stirred and left to cool, the spermaceti being removed when set, and the stearic acid thrown down from the aqueous solution by hydro-

chloric acid. On prolonged exposure to the air spermaceti becomes yellow and rancid, but can be rendered fit for use again by re-melting and treating with dilute caustic potash or soda lye. Spermaceti is scarcely affected by boiling with dilute sodium carbonate solution, differing in this respect from stearin.

The testing of spermaceti is performed (according to Kebler) as follows: The melting-point varies between 42° and 47° C., whilst that of cetin is between 48.9° and 55.5° C. The specific gravity at 15° C. is from 0.905 to 0.943, and is, throughout, not so near to the latter figure as formerly assumed. The saponification value ranges from 125.8 to 134.6, whilst the acid number exhibits wide fluctuations (0.0 to 5.17) as the age of the sample increases.

Uses of Spermaceti.—Medicinal: for salves, and formerly also as a palliative for alleviating pain and irritation; cosmetic: in pomades, etc.; technical: for candle-making (it burns with a brilliant, smokeless flame), either alone or in conjunction with wax; as an addition to dressings (for cloth), etc.

(f) *Whale Oil (Walfschithran).*—Under the name of whale oil there comes into commerce the oil from the blubbers of various species of whales (*Balæna*), the most important being the Greenland or Right whale (*Balæna mysticetus*), which yields also whalebone, the Sulphur Bottom whale (*Balænoptera sibbaldii*, Gray), the largest of all animals, being often 90 to 96 feet long and yielding 100 to 140 barrels of oil, the Hump-back whale (*Megaptera longimanus*, Rud.), 50 feet long and yielding 25 to 40 barrels of oil, inhabiting Southern seas, the Fin-back whale (*Balænoptera borealis*), which grows to 70 feet long, yielding 25 to 45 barrels of oils, and the Bottlenose whale (*Hyperodon rostratum*, Miller), 20 to 30 feet long, inhabiting chiefly the North Atlantic. There are many others, in all over 70 species.¹ Such oil has also been known as train oil,

¹ W. Mansbridge, *Jour. Soc. Chem. Indt.*, 1917, p. 362.

but this term is practically now restricted to the poorer classes of blubber oils of all kinds. As a rule the trade does not distinguish between the oils from various kinds of whales, although there are minor differences to be noted.

Whale oil is of a yellow, brown, or dark reddish-brown colour, its odour is strong and fishy. There are about five grades of oil distinguished in commerce. No. 0 and No. 1, very pale yellow, usually classed together; No. 2, deep yellow; No. 3, pale brown; No. 4, dark brown. Other darker qualities are classed as "dark whale oil" (Mansbridge). Its composition is not definitely known, but it contains valeric and soluble fatty acids. Crude whale oil deposits "whale or fish stearin" on cooling. This usually has a red colour, smells of fish, and consists of palmitin with a little spermaceti.

Whale oil is used in making soft soap, lubricating machinery and other purposes.

Other features of whale oils are mentioned below.

(g) *Fin-back Whale Oil*, from the *Balænoptera borealis*.—The oil is colourless to brown, and has a specific gravity of 0.920 to 0.925 at 15° C., according to class. It deposits a little stearin at 8° C., and thickens completely at 3° C. The dark kinds have a peculiar, highly repellent smell. With ether it is miscible in all proportions, and the darker grades manifest the remarkable faculty of mixing with equal volumes of alcohol to form a clear solution, quickly separating, however, into oil and an alcoholic solution, 10 parts of alcohol taking up 4 vols. of the oil. If now this liquid be mixed up again it remains turbid, separating again into the original volumes of oil and alcohol, the latter containing a little oil and stearin. In boiling alcohol nearly 4 parts are dissolved, and 4 vols. of the oil dissolve 1 vol. of alcohol.

Fin-back oil contains:—

77.05	per cent.	carbon
12.05	"	hydrogen
10.90	"	oxygen
100.00	"	

h. Greenland Whale Oil, from *Balena mysticetus*.—The carefully prepared oil is honey-yellow in colour, and smells and tastes like fish. Specific gravity at 15° C., 0.925 to 0.927; a few crystals are deposited at 10° C., which increase as the temperature falls, until, at -2° C., nearly all the stearin and a little spermaceti are down. Five vols. of the oil dissolve 1 of alcohol, and 10 vols. of alcohol 1 of the oil, but at boiling heat 2 vols. of alcohol dissolve 5 of the oil; it is also miscible in all proportions with ether. When heated for some time at 200° C. it decomposes and blackens. Its elementary composition is:

76.85	per cent.	carbon
11.80	"	hydrogen
11.35	"	oxygen
100.00	"	

The whale oils are neutral when fresh but become faintly acid after a while, and behave similarly to seal oils under the influence of various reagents.

Fuming nitric acid produces at first a brownish coloration with a very slight tendency towards blue, turning thereafter brown, and finally blackish-brown. Sulphuric acid (specific gravity, 1.65 to 1.70) colours them brown, subsequently blackish-brown (blood colour in the case of seal oils). Nitric and sulphuric acid in equal volumes give, when mixed with an equal bulk of the oil, a yellow coloration, turning reddish and subsequently a dirty brown. Caustic soda colours all fish oils red-brown, as does also syrupy phosphoric acid.

The various whale oils, all of which deposit stearin along with a little spermaceti, near the freezing-point, are further treated for the preparation of sundry commercial grades, which are principally obtained from Norway. The pure oils, *i.e.*,

pale, brownish-yellow to brown, are known as "unpressed whale oil" from the method of production, and are classified according to colour. When pressed below the freezing-point two different products are obtained "whale fat" or "train tallow" and "pressed whale oils," containing no stearin.

Whale oil has the following constants:

Specific gravity at 15° C.	0.9170-0.9225
Acid number	0.56-0.85
Saponification number	187.9-194.2
Iodine number	110.1-136.0
Reichert number	0.7-2.04
Hehner number	94.5
Refractive index, at 20° C.	1.4762
Unsaponifiable matter	1.37-3.40

2. SEAL OILS.

Raw Material.—The blubber of the finned mammals, the walrus, seal, or sea cow, in the northern and southern oceans.

Preparation.—The carcasses are brought to land and the skins removed and treated in a special manner. The blubber, which occurs as a more or less thick layer between the outer epidermis and the actual flesh, is then cut away as cleanly and carefully as possible, the entire sides thus obtained being placed in large recipients 9-11 yards long and nearly 9 yards wide, the bottoms of which are made of strong barks of timber and the sides of wooden posts set very close together, the oil flowing out through the interstitial spaces. Below these vessels is a somewhat larger stout wooden reservoir, only some 40 inches in height, for catching the escaping oil. Water is placed in the bottom, so that any leakage is detected without loss of oil, the water serving also to cleanse the oil from any admixture of blood. When the upper vessel is filled with blubber to a certain height the pressure of the mass commences to force out the oil, which then runs down into the reservoir below. The oil requires two or three months to fully exude, but the product is divided by drawing off the

oil at certain intervals, storing each lot in separate tanks, etc. The earliest runnings are pale, devoid of smell, and form the best quality "light train oil," the quantity being some 10 per cent. of the total 60 to 70 per cent. of oil obtained. In proportion as putrefaction progresses with the length of storage, and the cellular tissue is destroyed, the oil runs away more reddish-yellow in colour, the shade deepening continuously by reason of the spontaneous and progressive heat evolved in the mass, until finally the oil is a dark brown, and possesses a disagreeable smell and taste arising from the decomposition products evolved. When, after a lengthy period, the oil ceases to flow, the entire stinking mass is shovelled out and made up in smaller heaps, the oil thereafter escaping being collected as an inferior quality. The residue, still remaining is boiled in water in large iron pans, which, with the fleshy and other portions together, do not contain enough oil to run out spontaneously. During the boiling the oil ascends to the surface and is skimmed off, while the residue after drying, forms a good manure.

(a) *Seal Oil, Sea-Dog Oil*, from the common seal, *Phoca vitulina*.—In commerce there are two grades, the pale and the dark oil; it is thinner than the first seal oils, has a specific gravity of 0.9250, and does not deposit stearin until -2° C. is reached. One hundred vols. of cold alcohol dissolve 1 vol. of the oil; hot alcohol from 10 to 12 vols.; 10 vols. of oil dissolve $1\frac{1}{2}$ vols. of alcohol. The fresh oil does not show an acid reaction.

(b) *Archangel Seal Oil, Sea-Calf Oil*, from the *Phoca lagura*.—Colour pale yellow to brown; specific gravity at 15° C. = 0.9155 to 0.9165, the various grades having the following densities:—

Pale Archangel seal oil	at 15° C.	0.9165
Brown " "	" 25° C.	0.9170
Sea-calf oil	" 15° C.	0.9155

elephant, sea lion, eared seal, or New Holland seal, is pale yellow to brown in colour and has a specific gravity of 0·921-0·930 at 15° C.

All walrus and seal oils have a disagreeable smell, that of the brown kinds, which is due to putrefaction products, being the least supportable. They contain gelatin but no albuminoids; the former may be precipitated by treatment with pigments and metallic salts. The specific gravity at 15° C. ranges from 0·915-0·930; they are but slightly soluble in alcohol and require almost their own volume of ether to effect their solution. A few already deposit stearin below 5° C., others set only below zero C. (at -2 to -3° C.) to a solid mass. When quite fresh their reaction is but faintly acid, but the percentage of acid increases with age. They contain principally glycerides of phytetoleic, stearic, palmitic and a little oleic acid along with small quantities of butyric acid, valeric acid, etc.

Light-brown Greenland oil consists of :

77	to per cent. carbon
13·30	" hydrogen
9·40	" oxygen
<hr/>	
100·00	"

Seal oils vary in composition according to their origin and quality, the following figures having been recorded :—

Specific gravity at 15° C.	0·922-0·936
Solidifying point	2°-4°
Free fatty acids	0·2-24·00
Saponification number	178-196·2
Hellner number	62·8-95·85
Unsaponifiable matter	0·48-1·05
Refractive index at 20° C.	1·4816-1·4823
Reichert-Meissl number	0·96-1·66
Iodine number	124·2-193·3

Fuming nitric acid gives a red-brown coloration with all seal oils. Sulphuric acid (specific gravity, 1·65-1·70) gives at first a reddish-yellow, then reddish-brown, and finally brown-

red (blood-like) colour. Nitric acid and sulphuric acid—equal volumes—stain them a reddish tinge at first, turning to brown and finally dark-brown.

The tests for adulteration with other oils are directed to detecting other fish oils and rosin oils, which can be recognised on the one hand by their greater solubility in alcohol, and on the other hand by their incomplete saponification.

Marine Animal Oils.—An article in the *Bulletin of the Imperial Institute* (April-June, 1918) is devoted to a series of samples of marine animal oils which were prepared in Adelie Land by the members of the Australian Antarctic Expedition of 1912-13. These samples, thirteen in number, were forwarded by Sir Douglas Mawson, who is of opinion that an industry in these products is capable of being developed, provided that plans are first carefully considered and worked out before actual operations are commenced, and that some form of protection is devised to prevent the extermination of the animals. The samples, which were examined in the laboratories of the Institute, consisted of five samples of sea leopard oil, seven samples of Weddell seal oil, and one sample of penguin oil. These were extracted by different methods and from some the "stearin" had been removed: no doubt this could be done very effectually at such low temperatures as they must have been subjected to. The oils were all pale yellow or golden yellow in colour, except the penguin oil, which was brownish-yellow; they had one property in common, and that was their fishy odour. In the article referred to, analyses of all the oils are given, and the maximum, minimum and mean results are also brought together in a table which we reproduce below:—

<i>Sea-Leopard Oil.</i>					
	Sp. gr. at	15° C.	Saponification	Iodine	Solidifying
	15° C.	Value.	Value.	Value.	point of
					Fatty Acids
Max. . . .	0.925	191.5	127.5	119° C.	
Min. . . .	0.924	193.7	119.7	3°-4° C.	
Mean . . .	0.9245	194.4	123.8	—	

Weddell Seal Oil.

	Sp. gr. at 15° C. 15° C.	Saponification Value.	Iodine Value.	Solidifying point of Fatty Acids
Max.	0.941	201.5	147.8	19° C.
Min.	0.924	192.9	122.1	16-17° C.
Mean	0.9275	195.1	134.6	

Penguin Oil.

.	0.942	197.5	120.9	15° C.
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Several samples of these oils were submitted to a large firm of importers, who were of opinion that the sea-leopard oils were equal in value to Newfoundland seal oil, which in December, 1917, was worth £100 per ton, the Weddell seal oils would compete with No. 1 whale oil, valued at £55 to £56 per ton, while the penguin oil would be worth about as much as crude whale oil.

3. FISH WASTE TEIN OILS. FISCIPTERANE.

Raw Material. Small fish, such as the herring, sprat, sardine, sardella, pilchard, menhaden, or the waste materials therefrom, and from smelt, salmon, silurus, sturgeon, etc.

Preparation.—The heads, gullets and entrails of the herring, sardine, sardella, etc., as well as the whole fish of some kinds, when caught in excessive quantities and not otherwise utilizable—perhaps already in a state of putrefaction—are boiled with water in large iron pans, the fat collecting upon the surface being skimmed off, clarified in large vats, and packed for sale; the residue finds employment in the manufacture of fish guano. The use of sulphuric or hydrochloric acid has been found advantageous in boiling. In another process the fish are sprinkled with 5 per cent. (by weight) of a 45° Bè solution of ferric chloride or ferric sulphate, which preserves them unaltered for four or five days; they are then beaten to a pulp, and pressed, by which means a large quantity of water and oil is forced out. The press-cakes are easily dried, becoming friable and pulverulent, and

can be pressed a second time between hot plate, or extracted with volatile solvents, whereby a further quantity of oil can be recovered. Oil can also be obtained from such fish and fish waste by means of the centrifugal separator (German patent, No. 23,974).

The fish oils may be classified as follows:—

(a) *Herring oil, Swedish fish oil, Russian fish oil, Astrachan herring oil*;

(b) *Sprat oil*;

(c) *Sardine oil, Italian fish oil, Mediterranean fish oil*;

(d) *Pilchard oil, Spanish fish oil*;

(e) *Sardella oil*;

(f) *Menhaden oil*;

(g) *Salmon oil*.

Generally these fish oils have a specific fishy taste and smell, and are pale yellow to brown in colour, with a specific gravity of 0.925 to 0.930 at 15° C. Near freezing-point they deposit stearin. Alcohol takes up only 2 per cent. of the oil in the cold and 3.5 per cent. on heating, and 5 vols. of oil require 2 vols. of ether for their solution. All (waste) fish oils are browned by fuming nitric acid. Sulphuric acid of specific gravity 1.65 to 1.70 imparts a characteristic greenish coloration at first, turning to brown, and finally quite black. Sulphuric and nitric acid in equal volumes, mixed, produce at first a yellowish, then greenish, and subsequently brown coloration. Menhaden oil possesses drying properties and has been used for a paint oil.

Menhaden Oil.—The menhaden (*Alosa menhaden*) is a small fish of the herring tribe, about 8 to 14 in. long, which occurs in large numbers off the coast of North America. Recently a very interesting and full account of the menhaden industry, by J. W. Turrentine, appeared in the *Journal of Industrial and Engineering Chemistry* from which the following notes are taken:—

	Specific Gravity at 15° C.	Acid Number.	Saponification Number.	Haber Number.	Reichert Number.	Iodine Number.	Unsaponifiable.	Babco Refractometer at 25° C.
Menhaden (<i>Alosa menadieu</i>)	0.9284 0.9349	3.53- 11.74	180.7-195	—	11	147.9- 192.9	0.01-1.00	80.7
Cod	0.922- 0.928	11.9-39.4	180-195	—	—	13-160	—	—
Sardine. (<i>Clupea sardinas</i>)	—	4.6-41.7	—	35.60- 97.50	—	160.9- 191.7	0.48-1.01	—
Salmon. (<i>Salmo salar</i>)	0.9259	—	182.8	35.62	0.55	161.42	—	78
Herring. (<i>Clupea harengus</i>)	0.9178- 0.9391	2.4-41.2	179-190.5	35.64	—	103.4- 142	0.87-10.7	—
Sprat. (<i>Clupea sprattus</i>)	0.9274	—	194.5	91.4	Reichert- Veisli	122.5- 142	—	—
Pilchard or sardella (<i>Alausa pilchardus</i>)	—	134-198	185.3-186.4	—	—	150	—	at 40° C., 68.2-73
Japanese sardine. (<i>Clupeoides melanostictus</i>)	0.9316- 0.9347	10.8-34.5	189.8-190.2	94.5- 95.3	—	180.6- 187.3	—	at 40° C., 50.61

The history of the menhaden fishery is traced from before the advent of white settlers on the American continent, when the Indians of New England fertilised their crops by means of fish. Later it was found that the oil contained in the fish was of no value for manurial purposes, and that it could be readily removed. Hence rough and ready methods of extracting the oil were devised. The method most generally in use was to place the fish in barrels, along with water, and cover them with boards. In a few days putrefaction set in, the destruction of the tissues allowing the oil to escape and rise to the surface, from which it was removed by skimming. This method was also taken up by the white settlers, but in course of time it became superseded by the method of cooking the fish in pots, and submitting the mass to pressure, the result being a better class of oil, less offensive smell, and shortening of the time, besides which the fish scrap was obtained in a drier and better condition for handling. Later the method of cooking in pots was replaced by steam cooking, the first factory to adopt this process being a small one erected near Portsmouth, Rhode Island, in 1841. Since then the industry has gradually developed, hand presses introduced in 1856 giving way to hydraulic presses in the year 1858. In 1876 "floating factories" were introduced, these being equipped with boilers, cooking vats, and presses, but, owing to lack of storage room in them they were gradually abandoned.

In 1884 the business reached almost its high water mark, 858,592,691 fish being caught, yielding 3,722,927 gallons of oil and 68,863 tons of scrap. The smallest catch during recent years was in 1892, when 223,623,750 fish were taken. The average number taken during the last 30 years is approximately 500,000,000 fish. The incomplete figures for 1902 indicate, however, for that year 900,000,000 fish.

At present there are about forty factories on the Atlantic seaboard manufacturing fish scrap, most of them being in the

States of Virginia and North Carolina, the centre of the industry being the Chesapeake Bay region.

The fishing usually commences about April, and ends in November, the unloading of the vessels being mostly done by bucket elevators, which raise the fish on to the quay side, and deliver them into bins for the purpose of measuring. The fish, of course, vary in size, but it is considered that 1000 fish measure 22,000 cubic inches, therefore this bulk, whether it contains 500 or 2000 fish is counted as 1000; the weight is approximately 660 lb., and equals $3\frac{1}{2}$ barrels. From the measuring bins the fish are allowed to fall into storage bins, from which they are taken to the cookers.

The old method of cooking in open vats is still in use, but the newer method of cooking by steam is more expeditious, and, moreover, is continuous, the fish being fed into a long narrow cylinder provided with a screw for carrying them forward, while the steam is injected directly among the fish. The capacity of such a cylinder is about 100,000 per hour.

The modern screw press consists of a truncated cone placed horizontally; through the centre of this cone passes a shaft, on which is built a screw tapered to fit the cone-shaped curb. The rotation of the screw carries the cooked fish forward into the small part of the curb where it is submitted to pressure. The expressed material is forced through the slatted sides of the curb, the water and oil being caught by a metal shield surrounding the press, from which it is conducted by pipes leading to the oil room. The fish scrap passes out of the small end of the curb, and falls into the buckets of a conveyer, from which it is delivered to the drying-room. 100 lb. of the mass coming from the cookers contains 22 lb. of dry matter and 78 lb. of water. In the press 56 lb. are removed, leaving a mass of 44 lb., consisting of 22 lb. of dry matter and 22 lb. of water (50 per cent.). The pressing is both automatic and continuous.

Fig. 74 shows a battery of "Concentrator" drying machines constructed by Rose, Downs & Thompson, Ltd., for drying fish waste for conversion into meat meal or manure. The standard size of the cylinder is 13 ft. long by 5 to 6 ft. diameter. It is constructed either of steel or of cast iron and is jacketed for steam heating. The machine will hold $1\frac{1}{2}$ to $1\frac{3}{4}$ tons of offal which is dried in about eight hours.

The hot air drier for drying the fish scrap is almost universally employed. This consists of an insulated iron cylinder about 6 ft. diameter and 30 or 40 ft. in length. It is provided inside with a series of iron flanges, or shelves, about 8 in. wide, running the whole length. These are for the purpose of lifting the scrap and dropping it through the hot air. The cylinder is rotated by an electric motor, a forced draught of hot air being induced in it by means of a fan. The wet fish scrap is charged into one end, while the dry scrap falls out of the other into a brick chamber, from which elevators remove it to the bagging room. The transit of the material through the drier occupies from 3 to 20 minutes, in which time the moisture content is reduced to about 7 per cent. A million fish will produce 75 to 85 tons of dry scrap.

With regard to menhaden oil, it is stated that this oil appeared on the market in the early sixties in considerable quantities, and commanded very high prices. In the nineties the annual production exceeded 2,000,000 gals., a figure which has been closely maintained. The price in 1865 was \$1 40c. per gal., but it gradually fell to 17c. in 1905. The present price is 23c. to 28c. per gallon.

The mixture of oil and water running from the presses is conducted to the first or uppermost series of tanks, in which it is allowed to stand, the water and oil separating. This may be hastened by heating with steam, for which purpose coils are provided. The oil is allowed to flow by a suitable arrangement of weirs through a series of tanks in which by means of

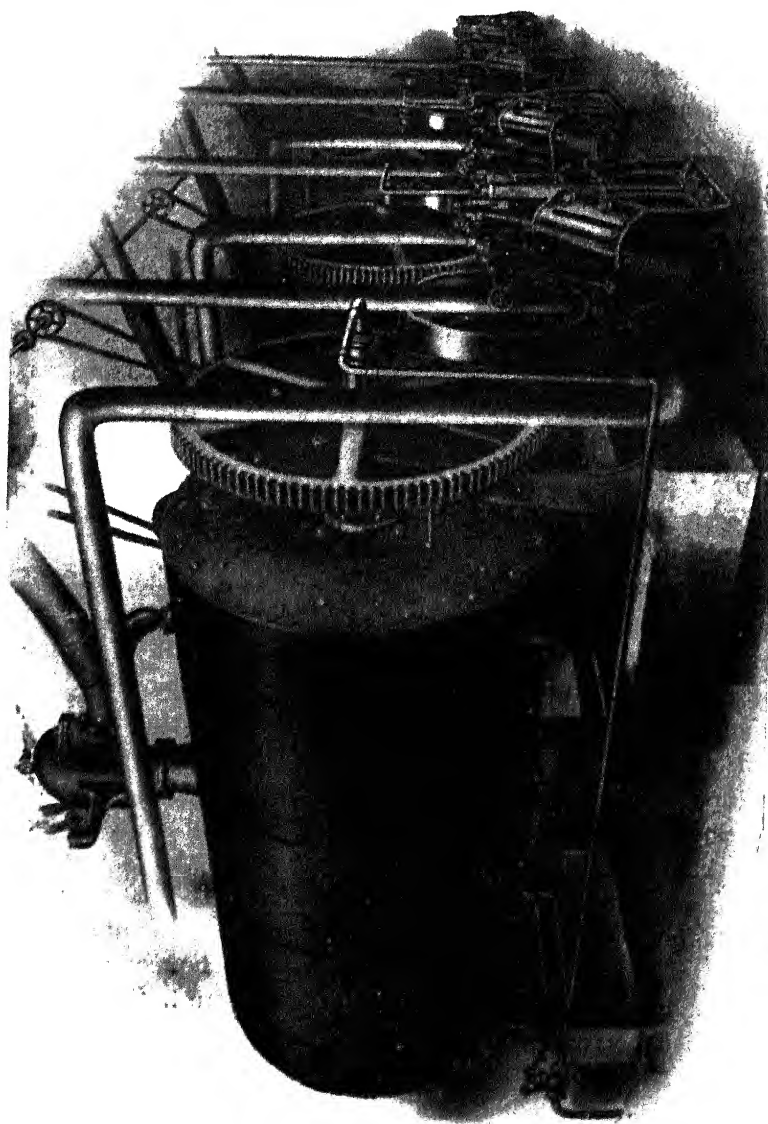


FIG. 74.—Battery of concentrator drying machines by Rose, Downs & Thompson, Ltd.

stronger heating by steam, it is gradually purified, the water and fine particles of fish settling out. The deposit, known as "gurry," is either sold to soap-makers or is pressed in bags for the recovery of the oil it contains. The purified oil is run into barrels.

The yield of oil in different years and seasons varies considerably. In the year 1900, the yield ranged from 3.51 to 6.39 gals. per 1000 of fish. The fish taken in the fall give the highest yield, *i.e.*, 12 or even 15 gals. per 1000 fish.

Crude menhaden oil varies in colour from light amber to dark brown. It was formerly used as an illuminant and for currying leather. It is used very largely as a paint vehicle, as a lubricant, and as a soap-making grease, as well as for tempering steel. It may be used as a paint oil, either mixed with 25 per cent. of linseed oil or along with a drier, preferably a tungate, *i.e.*, one made from tung oil by boiling with a lead or manganese compound.

The Fatty Acids of Menhaden Oil.—The determination of the composition of a mixture of fatty acids is not always an easy matter, therefore a paper by E. Twitchell in the same journal is especially welcome. The method employed was a modification of the well-known process of determining the molecular weights known as the freezing-point or Raoult's method. In this case the solvent is a pure fatty acid of known melting-point, which being one of the constituents of the mixture had no influence on the freezing-point, the depression found being due to the other acids present. It was assumed for this purpose that the fatty acids present in common fats, having about the same molecular weights, had practically the same influence, which is near enough to the truth, the depression in the freezing-point being 4° when 20 parts of one acid is added to 80 parts of another, used as solvent. The method is applicable only to solid acids, but it can be extended to the liquid acids by examining the solid products obtained by

hydrogenation; oleic, linolic, linolenic, and clupanodonic acid on hydrogenation all yielding stearic acid, and also to the products of fusion with caustic potash, unsaturated acids of the oleic series yielding solid acids with two less carbon atoms, oleic acid, for instance, being converted into palmitic acid, erucic acid into arachidic acid, etc. The members of the linolic series yield solid acids with four less carbon atoms, linolic acid, for instance, passing into myristic acid. With regard to acids with three and four double linkages, no experiments have yet been recorded, but they probably form acids with six and eight less carbon atoms respectively. A description is given of the methods of preparing pure myristic acid: M.p. 53.76, from nutmeg butter and pure arachidic acid, m.p. 74.78, from the erucic acid of rapeseed oil for use as solvents and the effect of adding 20 parts of one acid to 80 parts of another on the melting-point, the acids examined being behenic, arachidic, stearic, palmitic, and myristic. Full details are given of the method of determining the fatty acids in menhaden oil in the original paper. In a previous communication in 1914 the author gave the composition as far as it could be ascertained as:—

Palmitic acid	22.7	per cent.
Other solid, saturated acids	11.8	" "
Unsaturated acids with 16 carbon atoms	None	" "
" " " 18 " " "	26.7	" "
" " " 22 " " "	20.2	" "
Other unsaturated acids	18.6	" "
	100.0	

The figures are now:—

Palmitic acid	22.7	per cent.
Myristic acid	9.2	" "
Stearic acid	1.8	" "
Unsaturated acids with 16 carbon atoms	None	" "
Unsaturated acids with 18 carbon atoms = 26.7		
– 1.8 per cent. stearic acid	24.9	" "
Unsaturated acids with 20 carbon atoms	22.2	" "
" " " 22 " " "	20.2	" "
	101.0	

The fishery department of the Madras Government have been carrying out investigations into the production of sardine oil at their experimental station at Cannanore and later at Tanur, which has proved very successful. This has resulted in the founding of a new industry, there being in 1909 only one private factory in operation whereas at the beginning of the 1911-12 season there were some 40 or 50 small factories producing the crude oil in Malabar and South Canara, and similar developments were regarded as likely to take place also in Cochin and Travancore.

Several samples of the different grades of sardine oil extracted at the Government Station were examined at the Imperial Institute in 1912, the figures obtained for the yellow oils being as follows: Specific gravity at $\frac{100^{\circ}}{15^{\circ}}$ C., 0.876 to 0.878; acid value, 1.8 to 7.1; saponification value, 193 to 198; and iodine value, 154 to 159. Two crude brown oils had acid values of 35.0 and 53.5 respectively, otherwise the figures were the same. As the iodine values of these oils are practically the same as that of cod oil (about 155), which is used largely for leather dressing, it is suggested that these sardine oils would probably be suitable for the same purpose. The iodine value varies directly with the tendency to oxidise under favourable conditions, which latter is an important property in oils for leather dressing.

4. LIVER OILS (LEBERTHRANE).

Raw Material.—The livers of the following fish: Cod, ling, haddock, coal-fish, merlangus, or pollack, sea-pike, or small cod, shark (common shark, great shark, ice shark, hammer-headed shark), ray (prickly ray, smooth ray, spiny ray), skate.

Preparation.—The process of preparation is not universally the same, being carried out with greater or less care, so

that, naturally, the products differ very considerably with regard to colour, taste and smell.

1. After the catch the liver is cut out of the fish, collected, cleansed from blood and adherent entrails, and placed in tall upright casks fitted with three taps at the side and exposed to the sun. By the heat of the latter, and also that developed spontaneously in the mass, a liquid is obtained resembling poppy oil, known as "clear pale liver oil". This product is drawn off through the two upper taps, and the contents of the cask are then weighted with stones. The oil obtained from liver which has been long stored is never so clear and well flavoured as that from fresh. In the course of further spontaneous heating and the resulting putrefaction a further quantity of oil, known as "clear brown liver oil," is obtained, which is drawn off through the bottom tap. The residue in the cask is boiled or roasted in iron pans, yielding "brown liver oil".

2. By steaming the liver—a method calculated to advantageously replace the putrefaction process—finer and more limpid oils of milder flavour and almost neutral reaction are obtained, whereas the self-run oils are always decidedly acid. In the steaming process, the well-cleaned livers freed from blood and cut up small are treated with low pressure steam in closed vessels, whereby the tissues contract and the oil runs out. The product from the cod thus obtained is "medicinal (cod-liver) oil" or "steam liver oil," and being mainly prepared at Bergen in Norway, is met with in commerce as "Bergen cod-liver oil". In the same way also "Newfoundland" and "Labrador cod-liver oils" are prepared, these differing from the first named only by the somewhat larger content of stearin, which commences to deposit at 7° to 5° C. The residues from the steaming process are pressed, yielding a second, rather more highly-coloured oil. In the

preparation of liver oil by steam the following points have to be observed :—

(1) Great care must be exercised in the selection of the liver, only perfectly sound livers—but not green, reddish or blackish pieces—being chosen, since the coloured kinds specified are only fit for tanners' oil. Livers which have lain for more than twelve hours in summer or twenty-four hours in winter are no longer suitable for medicinal oil.

(2) Extreme cleanliness must prevail, and both the liver and the vessels used must be cleansed with hot water every time.

(3) The liver must not be heated above 70° C. nor exposed to that temperature for more than forty-five minutes at the outside; the shorter time it is heated the better is the resulting oil.

(4) Filtration must be carefully performed in order to remove even the very finest solid particles from the oil. During the entire process the oil must not be exposed to the air more than is absolutely necessary.

(5) Pressing is a method employed chiefly in Scotland. The fresh liver, thoroughly cleaned and cut up small, is heated in iron pans to 80° - 90° C., and continually stirred until converted into a pulpy mass, which is then placed in large calico bags, filtration taking place whilst hot, the residue being subsequently pressed for removal of the residual oil. At 15° - 16° C. the oils deposit a considerable quantity of stearin, which is removed by filtering. In other cases the well-cleaned and perfectly fresh livers are heated along with water to 80° C. until all the oil has exuded, the latter being skimmed off and filtered through flannel—a second filtration following in order to remove the solid matters which deposit on standing.

The percentage of oil yielded by the liver of the cod varies from 44 to $57\frac{1}{2}$ (Möeller). The "stearin" which separates on

cooling cod-liver oil is not like that of beef-steak oil, since it contains a large quantity of unsaturated substances, as the following figures by Heyerdahl show:—

Steam prepared oil	14.5
Oil freed from solid matter at 15° C.	14.7
Steam freed from oil by pressing	12.4

The following are the principal liver oils:—

(α) *Cod-Liver Oil* (Kablauthran, Dorschleberthran, Leberthran).

Of these there are many commercial varieties:—

a) *Clear Pale Liver Oil*.—Colour, golden yellow, odour characteristic, not disagreeable, flavour fishy, but not bitter; reaction faintly acid to litmus paper; specific gravity, 0.923 at 15° C.; solubility, 2.5-2.7 per cent. in cold, 5-6 per cent. in hot alcohol. Used chiefly for medicinal purposes.

(β) *Clear Brown Liver Oil*.—Colour, chestnut brown, smell and taste, strong, bitter and irritating to the throat, reaction, more strongly acid to litmus paper; specific gravity, 0.925 at 15° C.; solubility, 2.5-3 per cent. in cold, 5-6 per cent. in hot alcohol. Also used for medicinal emulsions.

(γ) *Brown Liver Oil*.—Dark brown, occasionally blackish-brown in colour, greenish to bluish-green by transmitted light, transparent in thin layers; peculiar, unpleasant, empyreumatic odour and bitter taste, highly irritating to the throat. The oil strongly reddens litmus paper, and has a specific gravity of 0.929 at 15° C. Alcohol dissolves 5.7-6.5 per cent. in the cold and 6.5-7 per cent. at boiling temperature; ether dissolves it in all proportions. Used under the name of "cod oil" in the leather trade.

It was shown by Heyerdahl that cod-liver oils contained very varying quantities of hydroxy acids, which were due to oxidation taking place during extraction. These hydroxy acids are very detrimental to the medicinal oil, hence the

heating of the livers and their exposure to the atmosphere is curtailed as much as possible (Möeller).

For ascertaining whether a "cod oil" is suitable or otherwise for use in the tanning industry, Liotman and Peters (*Jour. Socy. Chem. Ind.*, 1902, p. 693) use the following process: 0.5 to 1 gram. of the oil is weighed out into a flask of 400 c.c. capacity, and shaken well with 150 c.c. of water; 25 c.c. of normal permanganate solution is added and well mixed. The mixture is allowed to stand for two hours, shaking at frequent intervals. At the end of two hours the mixture is filtered from the black precipitate into a 250 c.c. flask and water added to make up to the mark. 50 c.c. were taken out and titrated with normal ferrous sulphate. A blank experiment is made at the same time, and the difference in the two titrations gives the amount of oxygen required to oxidise the oil. As the oxidation value increases the more unsuitable becomes the oil for leather making. Good oils give oxidation figures of 10 to 25; while those which are useless give figures ranging from 46 to 85. The iodine value, saponification value and acid value are of no service in determining the suitability of cod oils for treating leather.

Properties of Cod-Liver Oil.—These, so far as regards colour, smell, taste and solubility, have been already detailed. The elementary composition is as follows:—

75.91	per cent.	carbon
12.22	"	hydrogen
1.87	"	oxygen
<hr/>		
100.00	"	

Cod-liver oil is a mixture of numerous glycerides: those of oleic, therapeutic, jecoleic, stearic and myristic acids being the most prominent, also hydroxy acids or their glycerides; and, moreover, contains free acids such as butyric, acetic, gadoleic acids, and gall constituents such as fellic acid, cholic acid, bilifellic acid, biliverdin, bilifulvin. Among the bases

present are small quantities of trimethylamine, propylamine, amylamine, hexylamine, and a new base, hydrotoluidine, boiling at 198-200° C., as well as two non-volatile bases, aseline and morruhine (yellow oil, 2.5-3 per cent. ; white oil, 1.5-2 per cent. ; brown oil, 4.5-6 per cent. of morruhine). Most of these were the products of decomposition formed during the putrefaction of the livers and are not found in the medicinal oils of to-day.

Therapic acid was found by Heyerdahl to have the formula $C_{17}H_{26}O_2$ and to be a highly unsaturated acid of a new group, absorbing 8 atoms of bromine to form the compound $C_{17}H_{26}Br_8O_2$.

There are likewise present, in organic combination, small quantities of chlorine, bromine, iodine, sulphur and phosphorus compounds, along with the sulphates and phosphates of lime, magnesia and soda.

b) Cod-Fish Oil (Saythran, Sayleberthran, Kohlfischthran).—These oils correspond in flavour and smell with cod-liver oil. Cold alcohol dissolves 3.4 per cent., hot alcohol 6.5 per cent. The chemical composition agrees with the foregoing oil. Coal-fish oil exhibits the peculiarity of depositing solid fatty acids at 5-10° C., of which it contains, according to the researches of Kremel, twice as large a quantity as cod-liver oil; although it should not be forgotten that their amount depends on the more or less careful removal of the stearin during the preparation of the oil. The specific gravity is 0.925-0.927 at 15° C.

c, Shark's-Liver Oil (Haifischthran).—The liver of the shark is very heavy, often weighing as much as 1 ton (giant shark) and yielding some 5 cwt. of oil.

The colour of the oil is pale yellow and clear, and it still remains liquid when cooled to 6° C.; its specific gravity is 0.9105-0.9177, at 15° C., a shark's-liver oil with a higher specific gravity than this being always regarded with suspicion.

The smell is peculiar, but not very repellent, like the taste, which produces an irritating after-effect. Ten vols. of cold alcohol dissolve 1 vol., hot alcohol 4 vols.; 1 vol. of ether, 1 vol. of oil. The oil burns with a bright flame without carbonising the wick. The constituents are the same as in cod-liver oil, but in different proportions; the amount of iodine is somewhat larger.

(d) *Ray-Liver Oil* (Roehenleberthran).—Colour pale or golden yellow; taste and smell less unpleasant than in ordinary liver oil; it exhibits no acid reaction; specific gravity at 15° C., 0.928; cold alcohol dissolves 1.5 per cent., hot alcohol 14.5 per cent., cold ether $\frac{1}{2}$ vol., boiling ether 88 per cent. Near the freezing-point it deposits stearin; and it contains a larger percentage of iodine than cod-liver oil. Chlorine gas produces scarcely any alteration.

According to Benedikt, cod-liver oil is a highly complex mixture of small quantities of olein, physetolein, palmitin, stearin, etc., with the glyceride of a hitherto unknown unsaturated fatty acid, probably belonging to the linolic acid series. It contains variable amounts of free fatty acids, for the saturation of which Kremel found 0.62 to 28.67 grms. of calcium hydrate necessary per 1000 grms of oil. A little cholesterin (0.46 to 1.32 per cent. according to Allen and Thomson) is present which can be isolated by saponification and extraction with ether, the characteristic tabular crystals of cholesterin being obtained by recrystallising from alcohol the residue left after evaporating the ether. On the other hand, according to Jean, 6 per cent. of liver oil consists of a pale yellow, oily, unsaponifiable mass, which is stained a beautiful red by the addition of one drop of sulphuric acid.

Characteristic of this oil is its content of bile constituents, by reason of which several colour reactions can be obtained. The ash contains iodine, which can best be detected by saponifying the oil with caustic potash or soda, concentrating, calcin-

ing the residue, and then testing for iodine in the ordinary manner. None of the iodine can be extracted by shaking up with water or alcohol, so that the intentional addition of potassium iodide to this oil can be detected by extracting with alcohol and testing the residue left on evaporating the solvent. Kremel made the examination of liver oils the subject of exhaustive study, principally with the object of finding characteristic indications for distinguishing cod-liver oil, Japanese oil, coal-fish oil and seal oil.

As will be seen from the appended table, no reliable information can be obtained from the specific gravity, iodine number or saponification value. On the other hand, coal-fish oil contains twice as large a quantity of solid fatty acids as any of the other fish oils, which, however, may, as already mentioned, be due to insufficient separation of the stearin.

The following analyses were made by Kremel in 1884 :—

Oil.	Specific Gravity.	Percentage of		Melt- ing- Point of the Fatty Acids. ° C.	Acid Num- ber.	Saponi- fication Num- ber.	Iodine Num- ber.
		Liquid	Solid				
		Fatty Acids.					
1. Cod-liver oil, 1884		—	—	—	0.62	171	131
2.		92.12	6.72	—	1.41	171	127
3.		—	—	—	2.06	—	126
4. „ 1883		88.88	7.55	50.5	2.23	189	127
5.		—	—	—	2.32	—	128
6.		90.46	6.88	51	2.86	179	131
7. „ 5 years old	0.922	—	—	—	1.47	178	140
8. „ 10 „	to	—	—	—	28.67	—	—
9. „ 10 „	0.927	—	—	—	5.03	—	129
10.		—	9.60	48.49	9.59	173	139
11. Clear pale medi-		—	—	—	11.29	174	138
12. cinal liver oil		—	—	—	11.57	173	141
13.		92.72	5.25	52	8.66	181	—
14. Cod-liver oils from		87.00	12.75	51.52	6.78	181	135
15. 1883 and 1884		—	—	—	10.46	—	136
16.	0.925	75.32	19.04	55.56	1.26	177	137
17.	0.926	—	12.22	53	1.23	177	137
18. Coal-fish oils, 1883	—	—	—	—	1.29	179	129
19.	0.925	74.20	20.60	—	1.49	181	126
20.	0.927	70.00	21.34	52	11.8	181	123
21. Japanese liver oil	0.908	87.60	10.52	50.51	—	—	120
22. Seal oils, 1883	0.925	85.02	10.23	57.5	1.95	178	127
23.	0.925	88.29	9.81	57	2.01	179	128

The melting-point of the solid fatty acids is higher in seal oil than in the other kinds.

According to Mayer, a cod-liver oil is pure if, when shaken up with one-tenth of its bulk of nitro-sulphuric acid (1 : 1), it gives a coloration bright red at first but quickly turning to citron yellow, since in the other fish oils the transition is either not so distinct or on the other hand a brownish-violet coloration is produced.

Rössler agitates with aqua regia, with which pure cod-liver oil forms a greenish dark-yellow liniment, changing to a permanent brown after half an hour, whereas white seal oil or a mixture thereof with cod-liver oil gives a faintly yellow liquid under this test.

Kremel found the behaviour of the oil in presence of fuming nitric acid (specific gravity, 1.50) to be a far more suitable means of identification. If 10 or 12 drops of the sample be placed on a watch glass and 3 to 5 drops of nitric acid run in from one side, the following changes are observed :-

Pure cod-liver oil becomes red at the plane of contact of the liquids, turning to bright red on stirring, but quickly changing into a pure yellow.

Coal-fish oil becomes intensely blue at the point of influx, the colour changing to brown on stirring, and remaining so for two or three hours, finally becoming black.

Japanese fish oil behaves like the last named except that red streaks often accompany the blue.

Seal oil is unaltered at first, and turns brown only after some time.

The nitric acid reaction is so characteristic that the adulterations named can be detected easily in quantities from 25 per cent. downwards.

The adulteration of the oil with non-drying fatty oils can be detected by the iodine number, which is in these fish oils unusually high.

Drying oils may be recognised by spreading a sample out thinly on the surface of a glass plate, and keeping in a warm place; the fish oils, although they oxidise very quickly, form no solid skin.

If 1 part of fish oil be stirred up thoroughly with 2 parts of concentrated sulphuric acid in a tall glass, the mixture will be clear only in case no foreign fats are present.

Testing Fish Oils.—The better classes of fish oils are for the most part only adulterated with similar oils of low quality, falsification with other oils being practised on the medicinal oils only. In testing, regard must be paid to the specific gravity. Shark oil is the lightest, the specific gravity of this oil being 0.870-0.875 at 15° C.; next follow the sperm oils, 0.880-0.885; the whale (blubber) oils, 0.9140-0.925; the seal oils, 0.915-0.930; the liver oils, 0.920-0.950; and the fish (waste) oils, 0.925-0.930. These particulars refer only to the pale and clear brown oils, the darker kinds averaging about 0.005 more. A further guide to a decision is afforded by the varying solubility in alcohol.

Seal and whale oils are very readily soluble (the former up to 15 per cent.) in hot alcohol so that more than 1 vol. of oil dissolves in 10 vol. of alcohol, whereas liver oils dissolve only up to 6 or 7 per cent., and fish (waste) oils only to 4 per cent. By solution in alcohol the presence of resin and mineral oils can be detected at the same time, being characterised by their ready solubility in alcohol, but they require, however, to be confirmed by the saponification test, being themselves unsaponifiable. The addition of resin may also be proved by agitation in cold alcohol.

According to Salkowsky, the adulteration of liver oils with vegetable oils can be recognised by the following methods:—

The Setting-Point and Melting-Point.—The first of these is fairly low in fish oils, but the individual kinds vary considerably among themselves, owing to the removal of the

solid portions more or less by cooling. The time required for setting is also of importance, it being feasible in a few cases, viz., palm oil, coco-nut oil, palm-kernel oil, when something like 20 per cent. is present, to detect them by the rapid setting of the liver oil.

The Reichert-Meissl number was found for liver oils after deduction of the correction 0.2 (0.1-0.2 for the indicator per 5 grms.). For the majority of the fatty oils few higher numbers were obtained; only for coco-nut oil and palm-kernel oil were the numbers 7.38 and 3.48 found. Only these two oils would, therefore, be detectable with any degree of probability in fish oil, and then only when in large quantity.

Action of Sulphuric Acid.—The test is preferably applied in two forms, in that on the one hand sulphuric acid is run direct into the oil in a watch glass, and on the other, a few drops of the oil are dissolved in chloroform and then shaken up with the acid. A blue coloration ensues, the cause being attributable, not to the 0.3 per cent. of cholesterin, nor to any biliary colouring matter present, but rather to a pigment known as lipochrome.

Estimation of the Phytosterin Content.—The cholesterin of liver oil is not identical with that obtained from vegetable oils, the latter agreeing with phytosterin. Cholesterin sets to a pulp of laminated crystals, phytosterin to fascicular groups of solid needles. The melting-point of the latter is about 132°-134° C., that of cholesterin being 146° C. These differences are sufficiently characteristic to enable vegetable fats to be detected in liver oil. If 10 grms. of liver oil be saponified with 10 grms. of caustic potash and a little alcohol, then diluted with water to a 600-700 c.c. solution, shaken up with 500 c.c. of ether, and the ethereal extract filtered, evaporated and purified, if necessary, by a second saponification, the phytosterin will be obtained in a nearly pure state. That from liver oil will have a melting-point of 146° C., whilst that

from oil containing 20 per cent. of vegetable oils will melt at 139° - 140° C. The microscopic examination of the cholesterin affords another means of detecting adulteration (see p. 301).

Whereas liver oil contains, according to Allen and Thomson, 0.46-1.32 per cent., or, according to Salkowsky, only 0.3 per cent. of cholesterin, Jean found therein 6 per cent. of an oily, unsaponifiable substance, which gives a beautiful red coloration with a drop of sulphuric acid. In order to test the accuracy of these reports Fahrion determined the cholesterin in thirty samples of fish oils, by Hönig and Spitz's method. Of these, fourteen contained less than 1 per cent., eleven between 1 and 2 per cent., three between 2 and 3 per cent., and only two shark oils more than 3 per cent. of unsaponifiable matter. Shark oils are classed on the one hand with the liquid waxes, and on the other with the liver oils, the numbers obtained tending more in favour of the latter assumption.

In the case of cod-liver oils the darker sorts, as a rule, contain more cholesterin than the lighter grades. This may result from the method of preparation, the lighter qualities being obtained by cold, and the darker by the subsequent warm pressing. From the figures obtained it is permissible to conclude that the cholesterin content (6 per cent.) reported by Jean is quite possible.

The percentage of free fatty acids in good liver oils is very small, about 0.25-0.69 per cent., whilst most of the commercial vegetable oils contain larger quantities.

The method proposed by Salkowsky for estimating the volatile fatty acids insoluble in water is rather cumbrous and uncertain in its results and is, therefore, not very suitable for use in practice.

Dulière examined various liver oils and found the values given in the French *Pharmacopœia* to be too low; there the

Liver Oils.	Specific Gravity at 15° C.	Unsaponifiable.	Acid Number.	Saponification Number.	Reichert-Meißl Number.	Hehner Number.	Iodine Number.	Refractive Index at 15° C.
Cod. (<i>Gadus morrhua</i>) . .	0.9217-0.941	0.54-4.6	0.6-19.5	168.3-193.4	0.40-0.76	95.3-96.5	135-181.3	1.4800-1.4852 at 25° C.
Ling. (<i>Melca vulgaris</i>) . .	0.92-0.924	1.9-10.9	0.6	183.1-188.0	—	—	132.6-151.8	1.4747-1.4784
Haddock. (<i>Gadus aeglefinus</i>) .	0.929-0.934	1.1-1.3	0.5-2.7	187.2-193.0	—	—	154.2-186.4	1.4788-1.4806
Coal-fish merlangus or pollack. (<i>Gadus merlangus</i>) . .	0.9261	6.52	—	—	—	—	—	—
Shark. Various . . .	0.9105-0.9177	0.93-21.8	0.88-12.1	146.1-185.4	0.21	94.2	103.9-213.6	—
Ray. Various . . .	0.9161-0.9345	1.48-12.55	—	160.2-194.0	0.23-0.26	93.2-96	105.7-124.7	1.4752-1.4860
Skate. (<i>Squatina vulgaris</i>) .	0.9298-0.9307	0.97-1.08	0.75-4.86	184.5	—	94.7	157.3	1.4707
Japan fish oil . . .	0.916	0.6	31.2	—	—	95.3	90.0	—

specific gravity is given as 0.920-0.922, whereas he found it to be 0.9271 at 15° C. Oil prepared from fresh livers should not redden blue litmus paper. Contrary to the views of various investigators, Dulière was unable to discover any alkaloids in perfectly fresh liver oils or in the white commercial oils. The iodine numbers of liver oils freshly prepared by the author, as well as those of good commercial grades, approximate very closely, and range from 144-151; the Indian and similar oils have iodine numbers varying between 116 and 138.74, and that of Japanese oil is still lower, *viz.*, 98.74.

ARTIFICIAL TRAIN OIL AND ITS PREPARATION.

By artificial train oil is understood a dark-coloured product prepared from rosin oil and extensively circulated in commerce under the name of "lion train oil" (Löwenthran). It plays an important part in chemical industries on the small scale. The pale train oils, commercially known as fish oils, are as a rule pure, or at most adulterated, being rarely exclusively artificial products.

The dark oil is, as already mentioned, chiefly a rosin product. Although low quality materials alone are used for its production, the preparation is particularly difficult owing to the very particular requirements exacted by consumers. The criteria of the quality of artificial train oil are colour, odour, viscosity, and feel, as well, of course, it being necessary for the colour to be deep black, without fluorescence, by reflected light and reddish-brown in thin layers by transmitted light. The oil must be perfectly clear and of at least the consistency of syrup. When rubbed between the fingers it should not exhibit any great degree of slipperiness, although not sticky, such as happens when vulcan oil, for instance, is present.

It is a decidedly difficult matter to produce from the materials at disposal (in Germany); an artificial train oil

fulfilling the above requirements which can be sold at the low prices necessary to meet foreign competition.

The chief material employed is the so-called rosin stock oil, which in correspondence with the properties of the product, should be very thick and have the least possible odour and bloom. The paler and clearer the stock oil the better it is adapted for the purpose in view, a turbid and consequently darker rosin oil being much less suitable.

The next constituent in importance is "blue oil" (a crude petroleum product), the quality of which is even more particular than that of the rosin oil. The specially strong smell and fluorescence characteristic of Galician "blue oil" are generally so difficult to remove or mask that in its use the fundamental causes of bad quality in the final product are to be sought.

Unimportant as raw materials are the nitric acid and molasses, by means of which nearly all "lion train oils" are prepared. Two parts of rosin oil are mixed with 1 part of "blue oil" (the proportions being varied according to the degree of viscosity desired), a fairly concentrated nitric acid is then added which deblooms the mixture probably by the formation of nitro compounds from the hydrocarbons of the rosin oil, and, moreover, deodorises and darkens the product; 1.5 to 2 per cent. of acid is sufficient to effect these objects so far as they are possible with these raw materials. The smell of rosin oil in particular does not entirely disappear, but this does not so much matter, as in the finished product it will resemble train oil. The addition of 2-4 per cent. of molasses helps in producing the required colour, and more particularly in the masking of the odour. During the entire process the temperature in the pan—which is an open pan heated by direct fire—is kept at about 60°-80° C. As soon as the product exhibits the desired appearance it is left to stand for two or three days to clarify completely.

In the manufacture of leather grease, a large quantity of a liquid oil is recovered as well as the solid fat, which may be classed as "extracted train oil". This dark oil, which has a decided odour of fish oil and the properties of good natural fish oil, has yielded excellent results as an adjunct to artificial train oil; an addition of 5 per cent. of this leather-train oil to a comparatively poor "lion train oil" effected such an improvement in the latter that a dealer, who from long experience may be considered as an expert, and before whom samples of the original "lion" oil and the same after addition of the leather oil were placed for an opinion, classed the latter as good and the former as unfit for use.

In preparing light-coloured train oil the *modus operandi* is as follows:—

Good, pale, bloomless, faint-smelling rosin oil is mixed with $\frac{1}{3}$ to $\frac{1}{2}$ (according to the viscosity, etc., desired) of debloomed Russian oil of specific gravity 0.885 (or Galician oil of specific gravity 0.895-0.905) and warmed. When cold, 5-10 per cent. of pure fish oil is mixed in, together with a trace of trimethylamine solution, and well stirred. Should turbidity arise on account of the small quantity of water in the trimethylamine solution, which at most amounts to $\frac{1}{10}$ per cent., the oil is left to clarify. The product will resemble pure fish oil in all particulars, and although possessing a bitter taste, may be used with advantage as an adjunct to pure fish oil.

CHAPTER VI.

MISCELLANEOUS ANIMAL FATS.

EGG OIL.

THE yolk of the eggs of birds and reptiles contains a considerable amount of fat. Thus the yolks of the eggs of fowls have the following composition¹ :—

	Hen.	Duck.	Goose.	Turkey.	Guinea Fowl.
Water	49.5	45.8	44.1	48.3	49.7
Protein	15.7	16.8	17.3	17.4	16.7
Fat	33.3	36.2	36.2	32.9	31.8
Carbohydrates	0.4	—	1.1	0.2	0.6
Ash	1.1	1.2	1.3	1.2	1.2
	100.0	100.0	100.0	100.0	100.0

After evaporation the yolk of hens' eggs has the following composition :²—

Water	5.9
Protein	33.3
Fats	51.6
Carbohydrates	5.7
Ash	1.5
	100.0

For the composition of the yolk of turtle eggs see p. 248.

The egg yolk fat is obtained by boiling the yolks or by evaporation and hot pressing, also by extraction with solvents. The pressed oil contains only a small proportion of the lecithin

¹ U.S. Dept. of Agriculture, *Farmers' Bulletin*, No. 128.

² *Ibid.*

but the extracted oil contains a large quantity. The yield and composition of the extracted oil varies with the solvent used, thus Jean found that dried yolk yielded the following amounts: to petroleum ether, 48.24 per cent.; ether, 50.83 per cent.; carbon bisulphide, 50.45 per cent.; carbon tetrachloride, 50.30 per cent., and chloroform, 57.66 per cent. Vignon and Meunier recommend chloroform as a solvent and give the following results with ordinary hens' yolks:—

	Oil Per Cent.	Iodine Value of Oil.	Unsaponifiable Per Cent.	Phosphorus.	Lecithin.
Hen's egg yolk .	32.7	52	0.2	.737	21.0
Duck's egg yolk .	38.9	37.4	2.7	.604	17.3

The yolk fat from hens' eggs is an orange yellow semi-solid fat, and the expressed oil shows the following characteristics:—

Specific gravity at 15° C.	0.9144
Solidifying point	8°.10°
Melting-point	22°.25°
Saponification number	184.4-196.2
Acid number	1.2
Iodine number	68.5-81.6
Reichert-Meissl number	0.4-0.66
Hehner number	95.16
Refractive index at 25° C.	1.4713

Egg yolk oils examined by the reviser gave the following results:—

	Hens'	Ducks'
Iodine value	73.17-77.5	76.71
Saponification value	189.6-196.2	

Fat extracted by alcohol (lecithin) per cent.—22-27.36—

Saponification value	183.8-184	191.3
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Insoluble fat (triglycerides)—

Saponification value	190.4-193.3	190.4
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Fat extracted with benzene—

Insoluble in acetone (lecithin) per cent.	22.2	25.0
Soluble „ (glycerides) „	77.8	75.0

The fat in hen's egg yolk, extracted with ether, amounted to 30-33·26 per cent., and in dried yolk 45·2-49·5 per cent.

Yolk oil was employed in Russia at one time for the manufacture of soap (Kazan soap); it is at present employed only as a source of lecithin.

Egg yolk preserved with salt or boric acid is used for fat-liquoring leather, for which purpose it is extremely well suited as it not only contains a large amount of fat but it constitutes a valuable agent for the emulsification of other oils. Egg yolk is also used in embrocations and sometimes in margarine manufacture (see also p. 131).

TURTLE OIL.

Raw Material.—The eggs of the green and giant turtles of Jamaica and the Seychelles, and also those of the edible turtle. According to Brooks, it is obtained from the fat of these animals as well.

Preparation.—Turtle oil, or more correctly, turtle egg oil, is prepared by crushing the eggs with a forked wooden instrument in an empty bucket and pouring water on the mass, whereupon, after a few hours' exposure to the heat of the sun the oil collects on the surface of the water. Six thousand turtle eggs yield only 5 galls. of oil, and as 50,000 galls. of oil are annually dispatched to Para from the Orinoco, the Amazon and the Rio Negro, and 60,000 galls. are consumed by the native population preparing the oil, it will be evident that the turtles, which frequent the same sandbanks from year to year and lay 120 eggs apiece three times during the season, have extraordinary reproductive capacities.

The composition of the eggs of turtles is given as follows :—

	Fresh-water Turtle.	Sea Turtle.
Water	65·0	71·0
Protein	18·1	18·8
Fat	11·1	9·8
Ash	2·9	0·4
Undetermined	2·9	—
	<u>100·0</u>	<u>100·0</u>

When turtle oil is obtained from the flesh by boiling, the yield is uncommonly large, a single turtle often yielding 10 galls. of oil, so the output of 60,000 galls. at the Seychelle Islands is not very difficult of accomplishment.

In Jamaica, also, the oil is prepared from the flesh of the turtle, presumably from *Chelonia Cahanan*. Seychelle oil is superior in flavour, and might form a substitute for cod-liver oil, if its alleged properties could be substantiated.

The fat from the flesh of the turtle yielded the following figures on analysis:—

	Snapping Turtle. (<i>Trionyx sinensis</i>). ¹	(Thalassochelys <i>corticata</i>). ²	Green Turtle. (<i>Chelonia mydas</i>). ¹
Specific gravity at 15.5° C.	0.9229	—	—
„ „ 42.5° C.	—	0.9198	—
„ „ 15° C.	—	—	0.9335
Solidifying point	—	10°	—
Melting-point	—	23°-27°	—
Acid number	0.45	—	—
Saponification number	195.65	209	193.80
Reichert-Meißl value	—	4.6	—
Iodine value	121.09	112	127.4
Refractive index at 20° C.	1.4737	—	—
„ 30° C.	—	1.4677	1.4769

CANDLE-FISH OIL (GULACHONÖL).

Raw Material.—The candle-fish (“Gulachon” or “Ontachon”), found in enormous shoals in the bays on the coast of British North America and Alaska. This fish, which is also highly prized as a food, is so rich in oil that it is used in the dried state for torches; hence its name.

Preparation.—No information is available on this point, but probably by pressing like all other fish oils.

Properties.—Schædler gives the following description of its properties:—

¹ Tsujimoto, Lewkowitsch, *Oils, Fats and Waxes*, 1914, Vol. II, p. 464.

² Zdarch., *ibid*.

The oil as it is put on the market contains a considerable amount of palmitin and probably stearin, so that it is only semi-fluid at the ordinary temperature. The olein, or fluid portion, forms a clear, pale, straw-yellow liquid of fishy odour, differing from, and to many tastes being probably less obnoxious than, cod-liver oil. The sp. gr. is 0.9071 at 15° C. and 0.9012 at 25° C., whilst that of cod-liver oil varies between 0.92 and 0.93, but is generally 0.9227 at 15° C. When mixed with sulphuric acid (1 vol. of acid to 5 parts of oil) the temperature rises 55° C., whereas cod-liver oil under the same conditions undergoes a rise of 112° C. The colour reactions also differ from those of cod-liver oil, nitric acid (sp. gr. 1.27) mixed with one-third of its volume of the oil, producing at once a rose coloration gradually changing to amber yellow.

At the end of fifteen hours the mixture will have thickened considerably and assumed a deep amber coloration. Cod-liver oil treated in the same manner became rose-red at first, then rapidly changed to amber, and exhibited the same coloration in fifteen hours' time, but was more fluid than the first-named mixture and interspersed with thicker layers. Sulphuric acid gives with candle-fish oil not the same beautiful purple colour that it does with cod-liver oil, but a deep brown, subsequently inclining to both yellow and red. When this oil is saponified it yields 95.85 per cent. of insoluble fatty acids.

Candle-fish oil contains about 20 per cent. of palmitic and stearic acids, 60 per cent. of olein and 13 per cent. of an unsaponifiable substance, which, being a peculiar and interesting substance, deserves careful examination. At summer temperature this substance forms a mass of oily consistency, has a specific gravity of 0.865-0.872 at 15° C.—inferior to that of any other constituent of ordinary fats, and appears to be similar to the unsaponifiable matter in sperm oil.

CROCODILE OIL.

From the fatty portions of the reptile crocodile by rendering like tallow. Crocodile oil is of a reddish colour, thick fluid, peculiar odour, specific gravity, 0.928. Contains stearin, margarine, and olein. It is used in the tanning of leather.

ALLIGATOR OIL.

From the fatty parts of the reptile alligator of America by melting down like tallow. Alligator oil is of the consistency of ointment, semi-fluid, reddish in colour, with a specific gravity of 0.928, and contains 32 per cent. of palmitin and stearin, 1.5 per cent. of free oleic acid, 60 per cent. of olein and 0.02 per cent. of iodine. It is used in the tanning of leather.

HARE FAT (HASENFETT).

Hare fat is white, or occasionally somewhat yellowish, with characteristic odour and a mild, agreeable taste; the consistency is rather softer than that of lard, and it has slight drying properties.

Specific gravity at 15°	0.9349
Melting-point	44°-46° C.
Setting-point	28°-30° C.
Percentage of insoluble fatty acids	95.47 per cent.
Saponification number	200.9-204.9
Volatile soluble fatty acids (Reichert-Meissl number)	2.64
Iodine number	102.2-107.0
Refractive index at 40°	1.4586

Insoluble fatty acids:—

Melting-point	48°-50° C.
Setting-point	39°-41° C.
Neutralization number	209.0
Iodine number	93.3
Refractive index at 40°	1.4495

GOOSE FAT (GANSFETT).

Raw Material.—The accumulations of fat interspersed among the tissues and under the skin of the goose.

Preparation.—By carefully melting the cut up fat and straining to separate the product from the cellular tissue.

Properties.—It is white to pale yellow in colour, translucent, granular, frequently almost liquid at 10–12° C., of agreeable flavour and does not easily become rancid.

Specific gravity at 15°	0.9229–0.9400
Melting-point	27.5–34° C.
Setting-point (the temperature then rising to 22° C. (Schaedler))	18° C.
Saponification number	184.19371
Hehner number	92.4.95.88
Reichert-Meissl number	0.2.0.3
Refractive index at 40°	1.4593–1.4596
Iodine number	58.7.71.5

Uses.—For alimentary and medicinal purposes.

HUMAN FAT.

The investigation of human fat is of merely scientific interest, it will readily be understood that there is some little difficulty in investigating the product. Chevreul found this fat to consist of an olein and stearin, the latter differing from mutton stearin in yielding margaric acid instead of stearic acid. Heinz found, however, that the constitution was of a more complex character, and that in addition to palmitic and stearic acids other acids, one of them liquid, are present. The fat examined by Mitchell was freed from tissue at the lowest feasible temperature, was pale yellow in colour and somewhat softer in consistency than butter. When recrystallised from ether, only a small precipitate was produced, and this, after washing (with ether) and drying, melted at 51.5° C. Under the microscope the crystals exhibited some resemblance to those frequently obtained in the form of sheaves from lustrous

lard, the individual crystals exhibiting the characteristic chisel-shaped ends. An analysis of this fat gave :—

<i>Fat.</i>		
Specific gravity at 15° C.	. . .	0.9179
Melting-point	17.5° C.
Setting-point	15° C.
Saponification number	193.3-199
Reichert-Meissl number	0.25-2.12
Hehner number	93.92-96
Acid number	0.3 per cent. of oleic acid.
Iodine number	57.21-66.31

<i>Fatty Acids.</i>		
Melting-point	35.5° C.
Setting-point	30.5° C.
Iodine number	64
Iodine number of the liquid fatty acids	92.1

Twitchell's modification of the Jean method of fractional precipitation was employed for determining the nature of the fatty acids, 1.9478 grams of fatty acids (iodine number, 64) being dissolved in 25 c.c. of 95 per cent. alcohol and treated with 1.5 grams of lead acetate dissolved in 10 c.c. of the same alcohol, and the flask well corked and left to stand all night. Next morning a little of the liquid was filtered off, the acids being recovered by agitation with ether and hydrochloric acid, and their iodine number ascertained. The precipitate was well washed with alcohol, the fatty acids recovered, dried, weighed, and their iodine number determined, oxidation being in each case carefully guarded against.

The results were :—

	Grams.	Per Cent.	Iodine No.
Solid acids	0.9779	50.2	37
Liquid acids (by difference)	0.9699	49.8	92

The acids from the filtrate were still liquid at 15° C. ; those from the precipitate had a melting-point of 35.5° C. and a molecular weight of 278. From this may be calculated :—

Liquid acids in filtrate	99.8 per cent.
" " calculated as oleic acid	20.6 "
$\frac{37 \times 50.2}{100} = 18.57$	0.9
Total liquid acids	70.4 "
Iodine number, $\frac{64 \times 100}{70}$	90.9 "

Therefore, the liquid acids probably consist of oleic acid, with a small amount of a more highly unsaturated fatty acid, probably linolic acid.

So far as the solid acids are concerned, the high molecular weight of the lead precipitate (of which only 20 per cent. should be considered as liquid acids) leads to the conclusion that stearic acid or another high molecular acid is present.

The acids obtained by crystallisation from 70 per cent. alcohol melt at 50.5° C., which being a lower melting-point than is exhibited by any mixture of stearic and palmitic acids, indicates the presence of myristic acid. Human fat therefore consists of some 70 per cent. of liquid fatty acids, chiefly oleic acid, and 30 per cent. of solid acids, probably palmitic with small quantities of stearic and myristic acids and traces of lower volatile acids.

CHAPTER VII.

WASTE OR RECOVERED ANIMAL FATS.

DÉGRAS, TANNERS' GREASE, LEATHER GREASE, WASTE GREASE (GERBERFETT, LEDERFETT, ABFALLFETT).

THE substance indicated by the above names was originally a waste product obtained in the preparation of chamois (shamoy) leather, it was therefore not prepared originally on its own account. Formerly the price was high by reason of the article being a bye-product prepared in a special manner and only in limited quantity; and even now that dégras can be prepared in any desired amount, it is still saleable at relatively high rates, a circumstance rendering its production a profitable undertaking. In composition, dégras is really an emulsified fat, *i.e.*, a fat existing in an extremely fine state of division in a suitable liquid, so that it forms a mass of buttery or salve-like consistency. The operation by which it is produced is as follows: The skins to be dressed for chamois leather are unhaired by liming and scraping with knives, and then immersed in a bath of bran in a state of acid fermentation. They are afterwards wrung out, dressed over with train oil or cod oil, fullled for two or three hours and left exposed to the air a short time. These operations of fulling with fish oil and hanging out are repeated until the skin is full of oil and all the water has been expelled, the oil having meanwhile, under the influence of the air, become partly oxidised and intimately combined with the fibre. To complete this conversion, the skins, which are laid one above another, are subjected

to a kind of fermentation, air being admitted from time to time in order to prevent over-heating. The excess of oil is then removed by wringing, scraping, straking, or hydraulic pressing; and the skins finally treated with a lukewarm solution of potash from which further portions of the oil separate out on standing. According to another account the suitably prepared skins are sprinkled with fish oil and thrown into heaps which become fairly hot in consequence of the incipient decomposition of the fat; after a short time the skins are well fulled and finally treated with a 10 per cent. solution of potash, in which manner is formed the so-called white liquor, wherein the dégras, *i.e.*, the fat, is present in a very finely divided condition. To this liquor is added enough sulphuric acid to neutralize the potash, and when the liquid is left at rest the fat collects at the surface forming a dirty white, yellow or brown, mass of fairly mild flavour, principally used for softening tanned leather, which substance absorbs it with great facility.

In many factories hides are used for the sole purpose of manufacturing dégras, being shamoyed again and again by alternate oiling, fermenting, fulling, and treating with potash solution until they are practically worn away.

Various artificial products passing under the same name are used to replace true dégras, being prepared from fish oils and other fats, to which are frequently added tallow, rosin, oleic acid, etc., etc.

All kinds of dégras contain large quantities (mostly 12 to 20 per cent.) of water, which, in good qualities, should not separate out even after long standing. The emulsive capacity depends on the presence of a resinous oxidation product formed in the process. The larger the quantity of this substance present the more easily is an emulsion produced, a dégras containing 13.9 per cent. of resin substance and 53 per cent. of water producing such an intimate mixture with water

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The colour of this resin-like substance is brown, and its melting-point lies between 65° and 67° C. It is saponifiable, cannot be precipitated by common salt from alkaline solution (distinction from fats), is insoluble in water, soluble in alcohol and ether, but insoluble in petroleum spirit (distinction from rosin). It does not occur in fish oils, but is only formed in the process of shamoying. Most kinds of dégras contain unsaponifiable substances derived from the fish oil; thus, for example, dégras from sperm oil contains cetyl alcohol, and that from liver oil an oily, unsaponifiable mass, of which 6 per cent. is present in the unaltered liver oil. The content of free fatty acids varies greatly, but the quantity does not affect the quality of the dégras. Pure dégras is contaminated with solid organic matters such as particles of skin and membrane, which should, however, not exceed 5 per cent. The specific gravity of dégras, freed from water, *viz.*, 0.945 to 0.955, is higher than that of the fish oil used in its preparation.

An examination of fish oils, according to the method of Livache, by treatment with metallic lead, showed (in harmony with practical experience) that the fish oils best suited for the production of dégras are those most capable of absorbing oxygen. Whale oil is the most in demand, sardine oil, which yields only some 3 to 4 per cent. of resinous matter, being unsuitable.

EXAMINATION OF DÉGRAS.

Water Determination.—Five grams of substance are diluted with petroleum spirit and filtered through a tared dry tube containing a wad of cotton wool, the spirit being distilled off and the residue evaporated in a basin, dried at 120° C., and weighed. The insoluble portion is estimated by drying the filter tube at 120° C. and weighing, the contents being then

incinerated in a platinum crucible. If the residue is small in amount no addition of mineral matter has been made, on the other hand if large it must be weighed and qualitatively examined (for clay, chalk, gypsum, etc.).

Ash.—For this estimation 5 grams are taken. If the ash is alkaline, it is extracted with water, filtered and titrated with standard acid.

Mineral Acids.—If the dégras has a strongly acid reaction, 25 grams of the sample are boiled with 200 c.c. of water, left to cool, and the two layers separated by means of the separating funnel, the nature of the acid (mostly sulphuric acid) being ascertained from one part of the aqueous layer, while another aliquot part (50 c.c.) is titrated with caustic soda in order to determine the amount.

The following tests are performed on the dégras after drying and filtering through linen.

Unsaponifiable Matter.—A preliminary test by boiling with alcoholic caustic potash and decomposition with ammonia readily shows whether any unsaponifiable matters are present. If turbidity occurs the test is positive, a further examination is then commenced for the quantitative determination of the unsaponifiable matters, their investigation being subsequently carried on in accordance with the usual methods.

Resinous Matter.—The ether extracted soap solution used in the foregoing determination is evaporated until the solvent is expelled, and precipitated hot with an excess of pure sodium chloride. When cooled the coloured liquid is filtered from the deposited soap into a flask and the filtrate acidified with hydrochloric acid, whereupon the resin comes down in flakes, which unite on boiling and attach themselves to the walls of the vessel. After allowing to cool, the mass is brought into condition for weighing by agitation with ether, evaporation of the solvent and drying. Admixed resin is detected by extraction with petroleum spirit, which does not dissolve the resinous matter of dégras.

Extraneous Fats.—The presence of extraneous fats, such as wool fat, oleic acid, tallow, etc., may be suspected when the specific gravity of the oil extracted from dégras falls below 0·920, since dégras from fish and whale oils has a density of 0·949 to 0·955. For the presence of tallow, characteristic information is afforded by the melting-point of the fatty acids, those of tallow melting only above 40° C., and therefore raising the melting-point of those in the dégras, since the acids from whale oil melt at 24·9° C., those from liver oil at 18·5° C., and those from Japanese oil at 30·8° C.

Free Fatty Acids.—These are titrated by caustic soda and phenolphthalein. The saponification value 159 is used as a basis for calculating the percentage of free fatty acids in whale oil. The oil from dégras contains generally 15 to 19 per cent. of free fatty acids.

Jean gives the following examples illustrating the constitution of dégras :—

	1.	2.	3.	4.	5.
Water	18·9	14·84	12·93	28·9	19·20
Ash	0·25	0·13	0·55	0·7	0·07
Particles of skin	0·30	0·30	0·09	0·58	0·27
Oil	69·71	74·65	80·00	66·93	75·66
Unsaponifiable matter	6·84	6·05	—	—	—
Resinous matter	4·00	4·05	5·81	3·52	4·80

The following analyses are of curriers' and leather dressers' oils and of Sod oils :—

ANALYSES OF SOD OIL (JEAN).

	Per Cent.						
Water	16·40	26·300	36·18	19·45	11·37	13·74	10·54
Ash	0·135	0·219	1·48	0·62	0·65	0·26	0·28
Impurities	2·900	1·850	4·10	1·60	1·49	0·212	0·26
Unsaponifiable matter	17·430	0·167	traces	traces	0·53	0·27	—
Oil	35·070	50·610	19·07	29·83	86·49	84·095	88·92
Resinoid matter	0·900	5·519	12·57	13·32	1·28	1·423	3·24
Free fatty acids	27·200	15·300	26·60	28·00	6·7	—	—
Sulphuric acid	—	—	0·066	—	—	—	—

Fahrion, on the basis of numerous experiments, arrived at the conclusion that the same products are obtained from fish oil by shamoying as are yielded by oxidation in the air, and that the dégras expressed from shamoyed leather is nothing more than a partly oxidised fish oil mixed with larger or smaller quantities of nitrogenous matter extracted from the skin. Leinand's "dégras-former" is a mixture of oxyje-coric acids and their anhydrides, contaminated by small quantities of nitrogenous substances.

When dégras was introduced as an emollient into the tanning industry its use was found to be attended by so many advantages that the waste product of the shamoying process soon became insufficient to cope with the demand. In this extremity recourse was had to fulling not only raw skins but also the finished chamois leather with fish oil, exposing it to the air, and when sufficiently oxidised, recovering the oil by pressure as in the original process. Notwithstanding that the resulting product is identical as regards both appearance and action with the actual primary dégras (*première torse*), specially valuable properties are, in some quarters, still attributed to the latter. Against this view it must be emphatically asserted that, in general, the secondary products are preferable in so far that the fish oil extracts from the raw sheepskins the natural fat, rich in cholesterin, occurring therein, and at the same time takes up a great deal more of the nitrogenous constituents than in the case of the shamoyed leather. Fahrion prepared a similar product on a small scale by impregnating pieces of chamois leather with the sardine oil frequently employed, and, after three days' exposure to the air, extracting with ether. The "moëllon" thus prepared forms, when freed from water, a thick red oil. The analytical results obtained therefrom are subjoined, the figures for the original oil being added for the sake of comparison:—

	Fish Oil.	Moëllon.
Iodine number	193.3	75.4
Acid "	20.6	29.6
Unsaponifiable matter	0.6 per cent.	1.0 per cent.
Fatty acids	94.5 "	65.8 "
Total oxy-acids	0.7 "	2.0 "
Solid oxy-acids	0.2 "	7.3 "

The chamois leather has the same appearance after extraction as before, its only task in the *dégras* process being to finely divide the particles of oil and so render it more accessible to the influence of atmospheric oxygen.

From the sum of the unoxidised and oxy-fatty acids the presence of volatile matters in moëllon is revealed; since these are evolved by prolonged heating to 120° C., and at the same time, oxidation and condensation processes still go on at that temperature, the method of estimating moisture proposed by Jean (mixing with quartz sand and drying at 120° C.) must, therefore, be totally rejected. Drying may be continued all day long without the weight becoming constant, and almost any desired result may be obtained. Fahrion sums up the results of his extensive researches on the fish oils as follows: By far the most predominant solid fatty acid in fish oils is palmitic acid. Of liquid fatty acids they do not contain any of the known members—oleic, linolic, linolenic, isolinolenic acids—but, on the other hand, a hitherto unknown heptadecylic acid, asellic acid ($C_{17}H_{32}O_2$), and also—as a characteristic constituent which is the cause of their ready oxidation—the isomer of linolenic acid, jecoric acid, $C_{18}H_{30}O_2$. Very probably there are also other unsaturated fatty acids present; for example, the fact that in the combustion of oxyjecoric acid an excess of hydrogen is always found indicates the probable presence of a fatty acid isomeric with linolic acid. Nevertheless, since these other fatty acids do not conform to Hazura's law, nothing definite can be stated on the subject until further methods of investigation are discovered.

Preparation of Dégras from Fish Oil.—A considerable amount of dégras is prepared by a not very complex method, fish oil being converted into an emulsion in the following manner:

A 10-20 per cent. solution of potash is heated to boiling in a large pan, and fish oil is run into the hot liquid in a thin stream, from a vessel situated at a higher level, the two being kept continually in motion by means of a stirring apparatus. The quantity of oil employed must be determined for the various kinds of fish oil in each case; many kinds may be used up to 50 per cent. of the weight of potash solution, whilst with others the supply must be cut off much sooner. When all the oil has been added the paddles are kept at work for some time longer, and the mass is continuously heated, samples being taken from time to time, until, after prolonged standing in a tall test-glass, the milky, and apparently homogeneous, liquid does not separate into two layers, but only becomes thicker on cooling, whereupon the operation is considered at an end. The dégras is allowed to cool, but is still kept stirred, continuously, until it begins to thicken, when it is at once poured into the packages in which it is to be sent out for sale.

In the majority of instances the operation thus performed is conducted to a successful issue, a dégras conforming to all requirements being obtained. With some oils, however, all the trouble taken to ensure a usable product seems wasted. For example, the material may be heated and stirred, hour after hour, and the product will still, on standing, allow a great portion of the fat to separate out again, a circumstance leading to the conclusion that some kinds of fish oil are incapable of forming an emulsion with potash solution. On this account it is always advisable to subject oils intended for the manufacture of dégras to a preliminary examination to determine their behaviour in this connection; the test can be

performed in an enamelled vessel with a few quarts of potash solution.

Fish oils that will not emulsify properly with potash lye can, nevertheless, be easily worked up into dégras by first boiling a very weak solution of caustic soda in the pan, then stirring the oil in until the liquid is milky and adding a highly concentrated solution of potash, stirring incessantly until no more drops of fat can be discerned in the mass; the above described test is then performed. The operation is conducted as follows: In a pan of suitable size, solid caustic soda at the rate of 1 kilo (2.2 lb.) per 100 litres (22 galls.) of water is dissolved. Two vessels, one containing the fish oil, the other the concentrated potash solution, and both fitted with taps, are placed above the pan. The potash solution is prepared by mixing the potash with its own weight of water and leaving them to stand, with occasional stirring, for twenty-four hours, at the expiration of which time the concentrated liquor is poured off from the undissolved portion and placed in the vessel referred to.

The soda lye is first heated to boiling and the tap of the oil vessel then opened wide enough to allow a stream of the oil, of the thickness of a lead pencil, to flow, the liquid in the pan being meanwhile stirred with vigour. When the liquid has become milky after all the oil is in, the potash solution also is admitted in a thin stream, and the whole stirred until the sample shows a favourable result.

If it is desired to have a thick salve-like consistency, in the finished product, a somewhat larger quantity of caustic soda must be taken at the outset and the solution made of $1\frac{1}{2}$ -2 per cent. instead of 1 per cent. strength, the amount necessary being carefully ascertained by a preliminary experiment, since an excess of soda will make the cooled product more like soap than salve. The dégras should be of such a

consistency that it can be easily rubbed on to the leather by the finger and as readily absorbed.

Preparation of Dégras according to Herrburger. The methods of preparation are :

(1) 50 kilos of raw linseed oil, 1.25 kilos of fuller's grease (previously left to stand for four or five days over spent pine tan), 25 kilos of ordinary fish oil, 16 kilos of soft (potash) soap and 1 kilo of lime soap, perfumed with $\frac{1}{100}$ part of oil of valerian and $\frac{1}{2}$ per cent. of butyric acid.

2) A mixture is prepared from fish oil, a fish oil soap boiled with lime, and 100-120 per cent. of water, by shaking together until the mass sets.

(3) Fish oil mixed, according to requirements, with tallow and potash.

Preparation of Commercial Dégras. The white liquor from the shamoying process, or the oil obtained therefrom, and known as pure *dégras*, *molle*, or *moellon*, is only one of the constituents employed in commercial *dégras*; even the Paris makers, recognised as the best producers of *dégras*, only using it as an adjunct. When the waste product of shamoying no longer sufficed to supply the demand, factories were erected to treat skins with the sole object of producing *moellon*, soaking them in oil and then washing them with potash many times, until the skins were eventually completely destroyed. This product forms one constituent of commercial *dégras*.

For the production of the latter are required a copper pan, a wide wooden vat, a stirrer and a filter for straining. The materials are *moellon*, fish oil, wool fat, and commercial marble or freshly-burned white lime, the latter converted into hydrate by means of 30 Bé. caustic soda, for which purpose 50 kilos of soda lye are required per 100 kilos of white lime.

1. In the first place 80 kilos of fish oil and 2 kilos of marble or lime are placed in the pan (which must be large enough to hold double the quantity) and heated to boiling for

an hour, with continual stirring, in order that the two may combine thoroughly and the marble be prevented from subsiding to the bottom which if it occurred would give rise to charring, and thus render the oil dark coloured or black and unfit for use. The mass froths strongly; after the surface has become clear again the contents are passed through a fine silk gauze sieve into the vat and cooled down to 45° C. Meanwhile, 50 kilos of rain or river water are heated to 30° C. in the pan, along with the residue left in the sieve, and when the mass in the vat has cooled to the prescribed temperature, the water from the pan is run into it in small portions through the sieve, while stirring is kept up. When all the water has been incorporated, the stirring is continued until the mass is perfectly cold and it is then left at rest; the product will have the same degree of consistency as a soft butter.

On the following day 200 kilos of moëllon are stirred in, without heating, followed by a mixture of 100 kilos of wool fat, 50 of fish oil, 10 of tallow and 10 of palm oil, previously warmed to 40° C. in the pan and stirred until thoroughly intermixed. The whole is then kept stirred until perfectly cold, and at the end of twelve hours, by which time it will have attained a soft buttery consistency, it can be packed into the transport casks. If the water has been used too hot, or the above temperatures exceeded, the dégras will be quite fluid, water will separate out on standing and the mass soon become infested with mould.

2. Forty kilos of pale (*e.g.*, Greenland) fish oil are warmed and then mixed by stirring with

- $\frac{1}{2}$ kilo caustic lime from marble, and
- 15 kilos water, followed by
- 100 „ natural dégras,
- 10 „ light fish oil,
- 15 „ Lagos palm oil,
- 40 „ purified wool fat, and
- 30 „ water.

This dégras contains in percentages —

Pale fish oil, 20; mello dégras, 40; palm oil, 20; animal fat, 16; water, 18; and by reason of its content of soap and albuminoids it protects the leather from drying and preserves it for a long time.

Vienna's Method — A deep pan is selected for heating by direct fire; or, if steam be at disposal, a wooden vat, arranged for steam heating. In either case stirrers are provided these

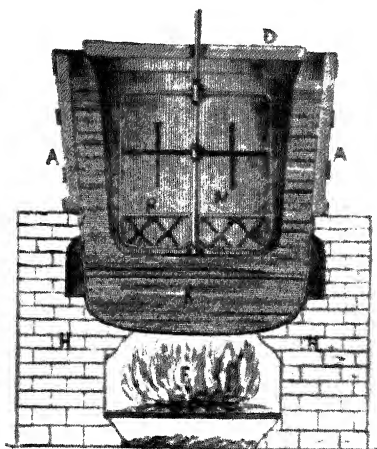


FIG. 75.—Pan, with stirrers, for preparing dégras.

appliances consisting of a vertical shaft (passing down the centre of the pan driven by gearing overhead), to which are attached several lateral arms, describing circles when the shaft is revolved, and keeping the contents of the pan in continual motion. When fire heat has to be employed it is advisable not to let the flames come in direct contact with the pan, but to surround the latter with a water jacket as shown in Fig. 75.

The pan K, mounted in the brickwork setting H, is surrounded by a wooden extension A, with which it makes a

watertight connection, so that by this means the pan M, wherein the dégras is prepared, can be placed in a higher position. R represents the stirrers, and D a wooden lid covering the pan M. By this arrangement the temperature in the latter vessel can never exceed boiling heat, and consequently the contents are prevented from charring.

A solution of 5 kilos (11 lb.) of potash in 10 litres (2·2 galls.) of cold water, prepared a day in advance by stirring up and then allowing to settle in a suitable vessel, is placed in the pan and 90 litres (20 galls.) of water are added thereto, the resulting weak lye being heated to boiling, and a few kilos of fish oil run in in the form of a thin stream with constant stirring. The heating and stirring are continued until a sample taken out is found, on cooling, to have the consistency of dégras.

The operation may also be modified by making an emulsion of fish oil with concentrated potash lye in the pan and then adding water until the proper consistency is attained.

Olein Dégras.—The olein or oleic acid obtained as a by-product in candle-making can be advantageously utilized for the preparation of a very useful dégras, which is, however, inferior in its properties to that produced from fish oil.

The mode of preparation is the same in both cases, the operation commencing with the running of the olein into the boiling caustic soda lye. The formation of the milky liquid is generally effected more quickly than when fish oil is used, and then follows the addition of potash lye and stirring until the product is finished.

If it appears from the quickly cooled sample that the material is still too liquid, a suitable quantity of olein soap is added to the (still warm) mass in the pan. In order that the soap may quickly dissolve without leaving any residue, it is essential that it should be cut into thin strips and the stirring continued until no white flakes of undissolved soap can be

detected in a carefully examined sample taken from the pan, the mass on the contrary appearing perfectly homogeneous.

Many practical men are of opinion that a brown-coloured *dégras* acts more powerfully than a pale one, a supposition that is nevertheless without foundation, the brown coloration depending merely on the use of dark-coloured materials in preparing the *dégras*. The demand for dark-coloured products can easily be met by the employment of a suitable colouring material added to the hot mass, a highly-concentrated extract of tan or tannin being the most suitable for the purpose. The liquid in question is run in a thin stream into the hot *dégras* and stirred until the colour is uniform; it is, however, necessary to see that the *dégras* preserves its salve like consistency. When spread out rather thickly over a glass plate and held up to the light, well prepared *dégras* should transmit the light uniformly without displaying any dark patches in the mass.

Dégras from Waste Fat. By suitable treatment any fat which, by reason of exceeding impurity, is scarcely fit for other purposes may be converted into *dégras*. Thus, for example, a very useful and high-priced *dégras* can be prepared from the dark and repulsive smelling fat obtained by boiling bones that are already in an advanced stage of putrefaction.

For this purpose the fat is heated in a pan for half an hour to 120° to 130° C., with continued stirring (whereby a very disagreeable odour is given off), and this is then stirred with 4 to 5 per cent. of its weight of a $\frac{1}{2}$ per cent. soda lye, followed by the potash lye as in the case of the *dégras* made from fish oil. In this manner can be obtained—generally without any soap being required for thickening—a brown *dégras* fulfilling all the requirements that can be exacted of the product.

Black dégras can be prepared by stirring into the hot *dégras*, coloured with tan or tannin extract, some green vitriol

(ferrous sulphate) dissolved in a small quantity of water, whereupon the entire mass will become deep black in colour. Such dégras forms an excellent emolient for black leather.

WOOL FAT (WOLFFETT, WOLLSCHWEISSEFETT, SEINT).

Raw Material.—The wool of the sheep, which contains a considerable quantity of this fat.

Preparation. By extraction with carbon bisulphide, petroleum spirit, etc., in the usual or specially constructed apparatus. The crude fat contains also decomposition products of gelatinous and albuminoid substances, to which the dark colour of the substance is due. If these products be successfully removed before they have undergone decomposition, a pale yellow wool fat is obtained, with gelatine and albuminoids as bye-products. With this end in view the best material to employ is the solution of wool fat obtained by extracting the crude fat or dissolving it in suitable solvents. This is treated with a small addition of syrupy phosphoric acid or one of the following precipitants: pyro- or meta-phosphoric acid, acetic acid, lead acetate, tannic acid—well stirred up and mixed without interrupting the stirring—with 2 per cent. of benzine, alcohol or strong spirit. In a very short time the precipitated substances will have settled out. The amount of phosphoric acid, etc., necessary for the precipitation varies with the origin of the wool and the content of fat, and must be ascertained beforehand, but as a rule it will not exceed 2 per cent. of the fat. The clear and now pale yellow coloured benzine solution separated from the precipitate resulting from the above treatment can be evaporated without delay, and will leave behind a pale yellow fat. If, instead of ordinary wool fat, neutral wool fat is desired, the solution freed from the precipitate is not at once evaporated, but is first treated with aqueous alkali, heated and well stirred, the aqueous layer containing the resulting soap being separated, after

a short time, from the supernatant solution of neutralizing. If the separation is attended with difficulty it is facilitated by the addition of a little alcohol or spirit. The line containing the neutral fat can then be evaporated, and is first treated with calcium chloride for the removal of articles of water and traces of soap dissolved therein in solution.

The separation of the gelatinous and albuminoid substances from the wool fat is easily effected in the above manner, provided the crude fat, or fatty matter from which the fat has been dissolved by benzine or the like, has not previously been subjected to prolonged heating. If, however, the fat in the benzine solution employed has already been heated to completely expel the solvent, or brown, residual wool fat obtained from crude wool fat by hot or similar methods is used, then the separation of the gelatinous and albuminoid matters by the aforesaid precipitation is incomplete, owing to the partial decomposition which takes place in the fat. The product, it is true, is paler than the original material, but will not yield a light-coloured residue. It is, therefore, necessary to lay particular stress on the selection of a material in which the gelatinous and albuminoid bodies have not suffered decomposition. The precipitation of the extraneous matters is best effected by the use of highly concentrated acids, such as the syrupy orthophosphoric or meta-phosphoric acids, glacial acetic acid, etc. Hydrochloric acid by itself will not produce any precipitate. The successive or successive action of the aforesaid precipitants on the dissolved wool fat, with the object of precipitating the gelatinous and albuminoid matters, is difficult, and it is necessary, after ascertaining the presence of such matters in the wool fat, to find the reagents suitable for their precipitation, since this cannot be deduced simply from general chemical data, the aforesaid precipitants being unable

by themselves to precipitate matters from solutions of wool fat, this result only ensuing on the addition of alcohol, which latter substance also, by itself, is incapable of effecting precipitation.

A considerable amount of crude wool fat is obtained as a bye-product from wool-washing works, especially in the Yorkshire district. The soapy liquors from the washing becks being run into brickwork tanks, where it is treated with a slight excess of sulphuric or hydrochloric acid. The fatty matters which rise to the surface are skimmed off and pressed in a steam-heated hydraulic press from which the melted fat runs into a cistern. The acidified liquid, still containing some fat in suspension, is run into the nearest stream. The press cake still contains 15 to 20 per cent. of fatty matter, and it would seem worth while to extract the cakes with a solvent; there is also present 2 or 3 per cent. of nitrogen so that they can be used for fertilising purposes. The crude wool fat contains in addition to the true wool fat a certain proportion of free fatty acids derived from the soap used in washing the wool. According to Lewkowitsch a grease prepared in this way had the following composition:—

Volatile acids	1.28 per cent.
Insoluble free fatty acids	20.22 „ „
Combined fatty acids	48.47 „ „
Alcohols (cholesterol, etc.)	36.47 „ „

This crude wool grease is employed on a large scale for oiling shoddy, it is also distilled and separated into an oleine and a stearine.

Properties.

Specific Gravity.—At 15° C., 0.973 (Schaedler).

Melting-Point.—39° to 42.5° C. (Stöckhardt).

Crude wool fat is a greasy, unpleasant-smelling, yellow or brown mass which, by reason of its high content of chole-

terin, isocholesterin, and their esters is only with difficulty saponifiable. Moreover, it contains, in addition, glycerides among which occur those of the lower fatty acids (*e.g.*, valeric acid) and potash soaps of the said acids. According to Darmstadter and Lifschutz wool fat contains lanoceric, lanopalmitic, myristic, and carnaubic acids, also two liquid and one volatile acid, ceryl alcohol, cholesterol, isocholesterol, oxy-cholesterol ($C_{26}H_{44}O_2$), and the ether of the latter ($C_{26}H_{44}O_2$)₂O. While Lewkowitsch states that it probably contains lower saturated alcohols and also some hydroxy acids. Altogether wool fat has a very complicated composition. When subjected to distillation it yields *distilled wool fat* which has the following characteristics:

Melting-point, 42° C.; setting-point, 40° C.; melting-point of the fatty acids, 41.8° C.; setting-point of the fatty acids 40° C.; saponification value of the fat, 169.8; of the fatty acids, 170.8; iodine number, 36.6 (Hubl).

Distilled wool fat consists almost exclusively of free fatty acids and cholesterol. When it is treated in the usual manner for the separation of the insoluble fatty acids a white mass is obtained, which, in a few days, becomes yellow and then orange, and, by reason of the presence of volatile fatty acids, resumes the characteristic smell of wool fat.

Purified wool fat is met with in commerce under the names of *lanolin* or *Adeps lanae* hydrous, containing about 20 per cent. of water, and *Adeps lanae* anhydrous. The crude fat, which under the designation of *Lisypus* was employed for medicinal purposes by the ancients, is a brown evil-smelling mass. According to Jaffé and Darmstädter the following means are employed for its purification. The soaps present in wool fat are converted into insoluble fatty salts by means of aqueous solutions of salts of the alkaline earths, or by metallic salts, and the mass treated in the centrifugal separator. It is then extracted by boiling acetone, wherein the wool fat

alone is soluble, and this substance is recovered by cooling the solution or distilling off the acetone. The crude wool fat treated with alkaline earths can also be dissolved in benzine, benzol, xylol, toluol, isobutyl alcohol, amyl alcohol, or carbon bisulphide, and the soaps thrown down by an addition of acetone. Instead of centrifugal force, lime water and an aqueous solution of a salt of an alkaline earth can be used for separating the wool fat from the wool washings. The precipitation of the lime soap breaks up the emulsion and causes the separation of the fat, which can then be skimmed off, and, when treated with alkalis, it can be made to yield up its fatty acids. When the products of the reaction are treated with methyl- or ethyl-alcohol lanolin is left behind as it is insoluble. Also in treating wool fat with alcoholic potash the pure wool fat remains undissolved. The wool fat is made into an emulsion with ammonia, and strong alcohol is added, whereupon the pure wool fat is precipitated; and it is this fat that, when kneaded with water, is put on the market as "lanolin". It melts at 40°C . and can absorb as much as 105 per cent. of water without losing its salve-like consistency. Being saponifiable with difficulty, and not becoming rancid, it is used as a basis for salves, an adjunct to cosmetic soaps, etc. A. von Rad removes the colouring and odorous matters of crude wool fat by treating it with oxidising agents, such as bleaching powder, or permanganate, in an alkaline solution, the wool fat being subsequently extracted with strong alcohol, benzine, carbon ether, benzol, tetrachloride or chloroform.

In order to obtain perfectly neutral products, wool fat is esterified in admixture with oleic acid, of which, however, no addition is really needed, since this acid is present in the raw material. This is treated with alcohol and sulphuric acid (66° B $\acute{\text{e}}$.). Rad designated the mixture of cholesterin ethers (wool fat) with the neutral ethers of oleic and ricinoleic acids

by the name of "lanesin". The *Norddeutsche Woll-kämmerei u. Kammgarn-spinnerei* prepares neutral wool fat by adding to the crude wool fat, thrown down from the wool washings by sulphuric acid, some fat solvent such as benzine or carbon bisulphide either before or after neutralization, following this up at once by a solution of common salt, magnesium chloride, sodium sulphate, etc. The soap is thereby separated from the dissolved wool fat, which can be recovered by evaporating the solvent. The use of sulphuric acid in this process may be superseded by sulphurous acid, which prevents putrefaction of the washings. The product so obtained is introduced into commerce under the name of *Adeps lanæ* for pharmaceutical purposes. J. Roos emulsifies wool fat with boiling water and soda solution, the layer of unsaponified cholesterin collecting on the surface being skimmed off and the residual emulsion treated with dilute acids for the recovery of the fatty acids, which latter are employed in soap-making under the name of "patent fat".

Purification of Wool Fat.—The preparation known as lanoline, now widely used in the manufacture of pomades and for other purposes, is a product extracted from wool fat and possesses the property of forming emulsions with water. The process of extraction and separation from the free fatty acids, odoriferous bodies, and other impurities of wool fat is effected in the following manner:—

An autoclave of some 1500 litres (330 galls.) capacity, consisting of a copper cylinder with well-riveted ends, is employed, fitted with a manhole in the upper part for the introduction of the charge of wool fat, and the other necessary materials for the process, the cover being screwed down whilst the apparatus is in use, and provided with a ring so that it may be removed when required.

Two steam coils are situated at the bottom of the autoclave, the one being perforated for blowing in steam in order

to raise the temperature rapidly and ensure the agitation and intimate admixture of the contents of the vessel, and the other closed, the function of this latter coil being to maintain constant temperature at the close of the operation, and so assist the separation of the soap from the etherified fat. In the middle there is a tap for decantation, and an outflow tap is provided at the base of the apparatus. In addition to these fittings the autoclave has a pressure gauge and a safety valve.

About 800 kilos (1760 lb.) of wool fat is placed in the autoclave along with a hectolitre (22 galls.) of caustic soda of 3.25° Beaumé strength, sufficient to saponify the free fatty acids. The manhole is then hermetically closed and steam is blown in for 3 hours at a constant pressure of 2 atmospheres. Into the mixture of soap and wool fat thus obtained are run 200 kilos (440 lb.) of water and 300 kilos (660 lb.) of acetic ether, the whole being then maintained in a state of brisk agitation by blowing in steam under 4 atmospheres' pressure during 8 hours. This operation results in the solution in the ether of the unsaponified wool fat, whilst the water absorbs the soap and the greater portion of the impurities.

On cutting off the supply of direct steam and keeping up the temperature by the aid of the closed steam coil, the liquid separates into two layers; the ethereal stratum—being lighter, and, therefore, at the top—is then removed by decantation through the upper tap, and washing with several additions of alkaline water at a temperature of 60° C. is resorted to in order to neutralize the acid liquor, the two final washings being effected with pure, warm water.

On distilling off the acetic ether, which may be used over and over again for an indefinite number of times, the wool fat is obtained as a white, neutral body, which, when filtered, may be employed for the same purposes as lanoline.

The water charged with soap is drawn off through the large tap at the bottom of the apparatus, and is collected in a lead-lined iron vat, where it is heated by steam. The vat is fitted with two taps, one about half-way up and the other at the bottom. This soapy liquor is treated with a 5 per cent. solution of 66° Bé. sulphuric acid in order to neutralize the alkali, and, after leaving to settle a while, the acid liquor is withdrawn and washing performed with pure water, maintained at boiling temperature by means of a jet of steam.

When the washings have become neutral, which can readily be ascertained by the aid of litmus paper, the fatty acids are collected and may be employed for soap-making, for which they are admirably adapted.

The acid liquors produced in these several processes are stored in reservoirs in order that the traces of fatty acids contained in them may ascend to the surface and be recovered by skimming, and the acidified water may be utilized in succeeding operations.

Lanolin Substitute.—Owing to the shortage of various materials the question of substitutes has become very prominent. Among the many things which are now difficult to obtain may be mentioned lanolin, the purified fat from the wool of the sheep. Lanolin is used in pharmacy in the manufacture of ointments, plasters, bougies, etc., for which purpose, owing to its unctuous nature, the ease with which it is emulsified, and its ready absorption by the skin, it is eminently fitted. In *The Journal of Industrial and Engineering Chemistry*, S. Axelrad describes his investigations undertaken with a view to finding an efficient substitute. The power which lanolin has to absorb water in such a high degree was ascribed by Liebrich to the esters of cholesterin which it contains, but Darmstädter and Lifschütz, after making a thorough examination of wool fat, came to the conclusion that this emulsive power was really due to alcohols of the iso- and

oxycholesterin group. A substance known as "Eucerin" was at one time imported from Germany, and Unna, treating upon ointment bases, states that this product is a mixture of alcohols of the iso- and oxycholesterin group in admixture with petrolatum. Unna, however, did not give any method for the preparation of these alcohols, nor did Roemer, who has also written upon the subject. Grim recommended the use of cetyl alcohol ($C_{16}H_{31}OH$) as a substitute for lanolin, and the author therefore investigated this substance in order² to determine its suitability. No satisfactory method for the preparation of cetyl alcohol on a commercial scale has yet been published, therefore attention was first directed to determine the cheapest and most practical method which would give the highest yield. After reviewing the methods which have been suggested, the author carried out a series of experiments, and found that the best process consisted in heating spermaceti with lime and a little water to a temperature of 230-250° C. At a temperature of about 240° C. cetyl alcohol distils almost unchanged in the form of a white vapour, distilling over to form an oil which soon crystallises to form a solid mass having a melting-point of 49.5° C., the yield being 40 per cent. on the spermaceti.

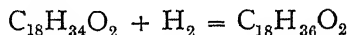
Mixtures were made of this cetyl alcohol with petrolatum, water, and a little lanolin, the following being found to be very suitable, and moreover showed no change after keeping for a period of 17 months: Petrolatum, 70 parts; paraffin (m.p., 60° C.), 20; cetyl alcohol, 10; lanolin (anhydrous), 5; water, 100 parts. The physical properties of this mixture resemble lanolin very closely, and it can be substituted for the latter in any ointment or other preparation. It is stated that the lanolin is employed to impart to the mixture the "unctuous" feeling which is so characteristic of wool fat; otherwise, it could be dispensed with.

CHAPTER VIII.

THE HYDROGENATION OF OILS FOR CONVERSION INTO SOLID FATS.

THE treatment of oily unsaturated fatty acids and their corresponding glycerides for the purpose of converting them into saturated substances of higher melting-point, and therefore of increased value, is a most important problem which has engaged the attention of investigators for quite a number of years.

Theoretically the conversion of oleic acid into stearic acid according to the equation—



seems to be a very simple reaction, requiring merely the addition of one molecule of hydrogen to each molecule of the acid to bring about the desired result, but on further acquaintance with the subject we shall find that in reality the problem is a very difficult one, which, though attacked time after time, has, until recently, yielded but indifferent results.

The passing of hydrogen through the liquid acids under any ordinary conditions leads to no change whatever; it is only by the application of special means that success has been attained, the number of researches which have been published and the host of patents taken out in connection with this subject, both in this country and abroad, being eloquent testimony not only of the difficulties met with, but also to the indomitable energy of those workers who have made this special branch of work their own.

The developments during the last few years have, however, been of such a nature that it is now not only possible to convert oleic acid into stearic acid, but to take ordinary oils, and in the course of a few hours, without decomposing them, convert them into solid fats. The importance of these discoveries to the makers of candles and soap cannot be over-estimated, but to others the subject is of equal interest, for it is stated that during the hydrogenation of fish oils, not only are they solidified but their vile taste and smell are removed so that they become fitted to be used for food and other purposes, and they now compete with the other more expensive solid fats.

The processes which have been devised for converting oleic acid into solid acids may for convenience be divided into three classes :—

1. Chemical processes.
2. Electrical processes.
3. Catalytic processes.

These processes are all fundamentally chemical, the only difference being in the means by which the change is brought about.

I. CHEMICAL PROCESSES.

The conversion of oleic acid into palmitic acid is possible by what is known as Varrentrapps reaction, *i.e.*, melting with caustic potash :—

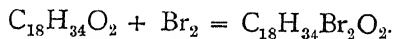


but as shown in the equation there is a considerable loss of material as acetate, and, though tried several times on a large scale, it has never been commercially successful.

By treatment of oleic acid with concentrated sulphuric acid, Geitel obtained sulphostearic acid ($\text{C}_{18}\text{H}_{33}\text{SO}_4\text{H}_2$) which on boiling with water yielded B. hydroxystearic acid ($\text{C}_{18}\text{H}_{35}\text{O}_3$) and sulphuric acid. Schmidt obtained a solid product (B.

hydroxystearic acid, m.p. 82° C.) by heating oleic acid with zinc chloride to 185° C., and then distilling in a current of steam for the removal of the oleic acid. This resulted, however, in the production also of a large proportion of stearylactone, and iso-oleic acid, together with unsaponifiable substances.

When oleic acid is treated with chlorine, bromine, or iodine the halogen is absorbed, forming, for instance, with bromine, dibromostearic acid :—



On submitting this compound to the action of zinc and hydrochloric acid, which liberate nascent hydrogen, oleic acid is reformed :—



If, however, the oleic acid be heated to a temperature of 200° - 210° C., with hydriodic acid and amorphous phosphorus, as proposed by Goldschmidt in 1875, then the reduction is complete, and stearic acid is the result. This constitutes the first successful method for the synthetic preparation of stearic acid. About fourteen years later P. de Wilde and A. Reyehler used chlorine, bromine, or iodine for the same purpose; for instance, heating oleic acid with 1 per cent. of iodine in autoclaves to a temperature of 270° - 280° C., and decomposing the product with a little soap solution.

The fat thus obtained had a melting-point of 50° - 55° C., and on distillation in a current of superheated steam was separated into stearic acid and other products. The conversion, however, was not complete, amounting to only about 70 per cent., but the difficulty of recovering the iodine was so great that the process was found to be too expensive to carry out on a commercial scale.

Treatment of the oil with chlorine for the formation of

monochlorostearic acid and subsequently saponifying the product was invented by Imbert (French Pat. 368,543 of 1906), while Zurer, after chlorinating, reduced the product with water and a finely divided metal (Ger. Pat. 62,407 of 1891).

According to a patent of Tissier (French Pat. 263,158, Jan. 16, 1897) oleic acid is reduced to stearic acid when it is heated along with water and metallic zinc under pressure in an autoclave. Oils heated under these conditions are stated to be decomposed into fatty acids and glycerine; at the same time the oleic acid is converted into stearic acid. Freundlich and Rosauer, however, after a number of experiments with oleic acid and with bone fat, were unable to detect any stearic acid to have been formed under these conditions.

2. ELECTROLYTIC PROCESSES.

The first claim to any method for electrically reducing oleic acid to stearic appears to be in an Austrian patent (No. 10,400, July 19, 1886) which was granted to Weineck. Ten years later Kness employed an electric current during the steam distillation of fatty acids with a view to increasing the proportion of solid acids therein. In 1900, Magnier, Brangier, and Tissier (Eng. Pat. 3363, 1900) proposed to acidify the oil material with sulphuric acid, and, after dilution with water, pass a current of electricity through the mass under a pressure of 5 atmospheres.

In the Hemptienne patent (Eng. Pat. 1572, 1905) claim is made for the use of a special apparatus consisting of a chamber in which a number of parallel metallic plates are arranged, these being placed alternately with glass plates. The metallic plates are alternately connected with the two poles of a generator, while the oil is sprayed on to the plates, and hydrogen is passed through under diminished pressure. By this method it was found that 20 per cent. of stearic acid could readily be produced; by repeating the treatment this was increased to

40 per cent., and after pressure to remove the liquid acid a further 20 per cent. could be obtained from the latter, leaving 40 per cent. of the liquid condensation products of oleic acid.

Using nickel electrodes, and passing the current through an alcoholic solution of oleic acid slightly acidified with sulphuric acid, Petersen was able to obtain a yield of stearic acid equal to 15 or 20 per cent.

The best results obtained by C. F. Bohringer & Son (Ger. Pats. 187,788 and 189,332, 1906) were, however, with electrodes constructed of a metal, for instance, platinum or palladium, on which a finely divided or spongy form of the same metal had been deposited. Here we approach closely to the purely catalytic methods, there being practically no dividing line.

3. CATALYTIC PROCESSES.

Catalytic agents are those substances which bring about chemical changes without undergoing any material alteration. This being the case, they are very often able, under favourable conditions, to do an amount of work altogether out of proportion to their weight, and at much lower temperatures than would be possible by strictly chemical means. The natural unorganised enzymes or ferments, such as diastase, ptyalin, pancreatin, etc., and the active principles of yeast, the butyric, acetic, and other living organisms, are all catalytic agents which induce changes in the surrounding mediums necessary to the life processes of the particular organisms which produce them, and at ordinary temperatures or at very moderate temperatures bring about most remarkable reactions with the greatest ease.

Certain inorganic substances, for instance, finely divided metals, metallic oxides, etc., have also been found to be specially active in certain ways, and to resemble the natural ferments in their action to a certain degree, therefore for this

reason they have been referred to by Sabatier as inorganic or "chemical ferments"---a designation which is not altogether misplaced.

The first recognition of the activity of an inorganic catalytic agent was made in 1820 when Edward Davy prepared platinum black and found that on exposure to air it absorbed oxygen so rapidly as to become red hot, and that on moistening with alcohol it caused the latter to take fire. The affinity of this finely divided platinum for oxygen is rather remarkable as no chemical action is observed and no oxide is formed; the action is simply a physical one, the oxygen being condensed on the surface and in the pores of the metal to such an extent that, as stated, the metal can absorb more than 800 times its own volume of the gas. This finely divided metal loaded with oxygen has now become a very active catalytic agent, but, as will be shown later, not only can platinum black absorb oxygen, but it can also absorb hydrogen or other gases, and thus its activity may in other directions be equally as powerful.

In 1822 Doberneier prepared spongy platinum, and found that when hydrogen was passed over it in contact with air the metal spontaneously heated up until the gas became ignited; this is the germ of many of the present self-lighting devices for incandescent gas burners. Later, Coquillon showed that when palladium foil (a similar metal to platinum) was put into a mixture of hydrogen or other combustible gas and oxygen, combination at once took place without explosion. Spongy palladium has since been produced, and also palladium black, which is still more active.

Other finely divided metals have been found to have active properties, as, for instance, "spongy" iron, which was used by Bischof for the filtration of water, and finely divided lead, which is pyrophoric, spontaneously taking fire immediately it is brought into contact with the air.

In 1871 Saytzeff found that nitrobenzol could be reduced to aniline by passing it in the form of vapour along with hydrogen over palladium black.

In 1901 Senderens obtained a patent (Ger. Pat. 139,457, of 1901) for the same purpose, but employing heated metals, for instance, nickel, copper, cobalt, iron or palladium, and hydrogen or water gas.

This is an interesting patent because for the first time other metals besides platinum and palladium, notably nickel, are recognised as catalytic agents. It was followed by a considerable amount of work by Sabatier and Senderens upon the action of various catalytic agents, which work forms the basis of our present knowledge of these remarkable substances.

During the years 1901 to 1904 several papers by Sabatier and Senderens appeared, these being devoted to the reduction of nitrobenzol by means of finely divided copper; the synthetic preparation of marsh gas (CH_4) from carbon monoxide and dioxide by means of finely divided nickel; here cobalt was found less active, and platinum, palladium, and iron are stated to have no reducing action; direct reduction of nitro-derivatives and oxides of nitrogen by finely divided nickel and cobalt; conversion of aldehydes and ketones into alcohols by hydrogenation, and direct hydrogenation of aniline and its homologues by means of nickel.

In subsequent years Sabatier branched out into the production of illuminating gases, taking out several patents for the formation of marsh gas and other hydrocarbons from carbon monoxide and other gases.

In 1907 Sabatier and Maille investigated the action of finely divided nickel and cobalt on the fatty anhydrides (acetic, propionic, butyric, etc.), also on alcoholic quinones.

Eventually Sabatier took out a patent (Fr. Pat. 394,957, Dec. 12, 1907) for the catalytic conversion of liquid into solid

fatty acids, which, however, was not the first one granted for this purpose.

In the meantime Senderens passed on to the investigation of the catalytic action of alumina, thoria, and similar earths on organic acids, ethers, ketones, etc.

Interesting as all these investigations were from—shall we say a catalytic point of view?—they are mostly outside the scope of the present article; therefore, we must go back a few years when we find that the new ground opening up was rapidly taken by other observers, the knowledge gained being applied to solving other problems than those apparently foreseen by the original investigators at that time. Naturally, the reduction of oleic acid to stearic was one of the problems which soon suggested itself, and we find the first patents taken out in this direction to be one granted to Le Prince and Sieveke (Ger. Pat. 141,029, Aug. 14, 1902), followed the next year by a patent to Normann, of Westphalia (Eng. Pat. 1515, Jan. 21, 1903). In the latter the fatty acid, or glyceride, either in the form of vapour or as a liquid is brought into contact with hydrogen or a gaseous mixture containing hydrogen, *e.g.*, water gas, in the presence of a finely divided metal such as iron, cobalt, or preferably nickel. The vessel used is a cylindrical one, filled with a catalyst consisting of nickel reduced from the oxide by means of hydrogen, and for the purpose of exposing as large a surface as possible this is precipitated on a support consisting of pieces of pumice stone.

By passing a strong current of hydrogen through a mixture of oleic acid and catalyst for a considerable time it is stated that the former is completely converted into stearic acid, while tallow treated under the same conditions has its melting-point raised to the extent of 12° C.

In 1906 Fokin, experimenting with ethereal solutions of oleic acid and catalytic metals, was able to obtain states that, on treating an ethereal solution of

oleic acid with palladium black and passing hydrogen through, stearic acid could be detected at the end of half an hour, with platinum black, 24 per cent. of stearic acid could be obtained in half an hour, $84\frac{1}{2}$ per cent. after $3\frac{1}{2}$ hours, and 90 per cent. after 5 hours. With cobalt hydride at 270° under atmospheric pressure the reduction of oleic to stearic acid was equal to 26 or 28 per cent., while in a sealed tube at higher pressure 60 per cent. was obtained. Nickel and cobalt (prepared from the oxides) also reduced oleic acid in presence of hydrogen, the former at 45° - 184° C., the latter at 98° - 250° C.

Fokin regarded the reduction, whether electrolytic or otherwise, as due to occluded hydrogen; therefore those metals which occlude hydrogen in the highest degree are most efficient. Palladium was found to be the most active metal, reduction taking place at 180° - 200° C., nickel, cobalt, and copper in the order of activity. The investigator suggests the "hydrogen" value of oils as the number of c.c.'s of hydrogen measured at 0° C. and 760 mm. pressure, absorbed by an oil in contact with molecular palladium, as an analytical factor equivalent to the iodine value, with which it agreed very closely in several instances.

Schwoerer (Ger. Pat. 199,909, 1906) claims the use of a close steam-jacketed pan containing a helical trough, over which the material to be hydrogenised is caused to flow in a thin stream. The catalyst, consisting of nickelised asbestos, is attached to the underside of the trough. Oleic acid, atomised by a jet of steam in presence of hydrogen, is blown into the vessel, which is kept at a temperature of 250° - 270° C. The vapours of oleic acid passing from the trough encounter the catalyst, and are thereby reduced.

In Erdmann's patent (Ger. Pat. 211,669, 1907) the oil in the form of a spray is dropped by a sprinkler on to a layer of the catalyst, which consists of nickel, supported on pumice or

the like. The temperature of the chamber being kept at 170° - 180° C.

In a modification of this process a tower partly filled with the catalyst is employed, and it is proposed to distil off the saturated product from the reaction chamber under diminished pressure.

In the Testrup process (Eng. Pat. 7726, 1910), unsaturated fatty substances mixed with a catalytic agent such as finely divided palladium, or preferably nickel (the latter 2-3 per cent. on the weight of the oil), is introduced in the form of a fine spray into a long cylindrical-jacketed chamber, heated to 160° - 170° C.; the chamber being fitted with a stirring device. From the first chamber it is taken by means of a pipe and sprayed into a second similar chamber, heated to 100° C., where the reaction is completed. If harder products are required the process is repeated, or the material is passed through several chambers.

Bedford and Williams (Eng. Pat. 29,612 of 1910) claim the method of preparing hydroxy fatty acids, or their glycerides, by using a pan heated with a steam coil, in which the material is placed along with a metallic oxide catalyst (preferably nickel oxide, added in the proportion of about 1 per cent.) and both hydrogen and oxygen passed through.

Schukoff (Ger. Pat. 241,823, 1910) also introduces very novel matter in his patent. This is for hydrogenating oil with finely divided nickel produced from nickel carbonyl. Carbon monoxide is led over nickel at the appropriate temperature, and the nickel carbonyl thus produced is led into the oil to be hydrogenised at a temperature of 180° C. As soon as sufficient of the gas is absorbed the temperature is raised to 220° - 240° C., whereby the carbonyl is decomposed, yielding nickel in a very finely divided state. At this stage hydrogen is led in, which accomplishes the reduction.

M. Wilbushwitz (Fr. Pat. 426,343, 1910) claims the treat-

ment of the oil with a catalytic agent in cylindrical autoclaves with conical bottoms. The oil is blown in at the top in the form of a spray, meeting an upward current of hydrogen. It is withdrawn by a tube and blown into the second chamber, and so on through a series. The pressure in this case is high, *e.g.*, nine atmospheres, and the temperature 100–160° C.

The catalytic agent may be prepared by dissolving iron, copper, nickel, etc., in an acid, and mixing the solution with twice its weight of a solid inorganic substance. The metallic salt is then converted into carbonate, ignited to oxide, and reduced in a current of hydrogen in a rotary drum at 500° C., the metal enveloping the inorganic particles. Claim is also made for the recovery of the spent catalytic agent.

On the same date Crossfield and K. E. Markel were granted a patent (Eng. Pat. 10,782, 1910) for the preparation of nickel catalysts in which kieselguhr, asbestos, pumice, and the like substance are impregnated with a solution of nickel sulphate, and the product treated with an alkali hydroxide to precipitate nickel hydroxide upon the porous material; after well washing, the substance is dried and reduced. If kieselguhr is employed the finished product should contain about 40 per cent. of nickel.

The Vereinigte Chemische Werke A. G. (Ger. Pat. 230,488, 1910; Eng. Pat. 18,042, 1911) prefer to use a palladium catalyst.

This is precipitated upon a metal which has no anticatalytic properties, or upon a metal oxide or carbonate. The hydrogen is used under a pressure of 2–3 atmospheres. Under these conditions it is affirmed that 1 part of palladium suffices to convert 100,000 parts of an oil into a solid fat in the course of a few hours. Certain gases or vapours were found to have a marked action on the catalyst, entirely preventing its action. These are arsenic, hydrogen phosphide, liquid hydrocarbons, carbon bisulphide, chloroform, acetone, and free mineral acids.

In a subsequent patent (Fr. Pat. 434,927, 1911) the same firm claim the use of solid salts of the metals of the platinum group for hydrogenating oils, the materials being heated to 100° C. in hydrogen, preferably at a pressure of 2-3 atmospheres. The reaction is stated to be quicker than with palladium or platinum black; while the activity of the salt is such that a quantity of palladium chloride equal to 1 part of palladium will convert 50,000 parts of an unsaturated oil into a solid fat. To guard against the liberation of free fatty acids by the hydrochloric acid formed by the dissociation of the salt, carbonate of soda may be added to the salt of the metal.

For the preparation of catalysts which are stable in the air, Kayser (U.S. Pat. 1,001,279, 1911) reduces oxide of nickel or equivalent metal in a current of hydrogen, subsequently passing carbonic acid through the reduced metal until it has cooled to the atmospheric temperature.

In the second patent (U.S. Pat. 1,004,034, 1911) Kayser claims the preparation of a catalyst by saturating kieselguhr with a concentrated solution of nickel sulphate, or other nickel salt, leaving it in an apparently dry condition. This is then mixed with the molecular quantity of sodium carbonate, thrown into boiling water, dried, and reduced.

H. Schlink & Co. (Eng. Pat. 9147, 1911) hydrogenate oil by passage through a centrifuge, the drum of which carries a lining of palladium catalyst.

K. H. Wimmer and E. B. Higgins (Fr. Pat. 441,097, 1912) employ organic salts of the catalytic metals. For instance, 100 grms. of cotton-seed oil are mixed with 1.5 grms. of nickel formate at a temperature of 170°-200° C., in hydrogen under pressure.

Ellis has patented three forms of apparatus for the hydrogenation of oils (U.S. Pats. 1,026,156, 1,040,531, and 1,043,912, of 1912). In the first the catalyst is placed on trays or baskets, the oil travels in a cyclic path downwards through

several layers of the catalyst, while hydrogen passes in the opposite direction. In the second patent a closed conical jacketed pan is employed, the oil and hydrogen being brought into intimate contact by taking the gas from the top of the chamber and pumping it in at the lower part of the cone, where it bubbles up through the mixture of oil and catalyst—a process which is automatic, and can be continued as long as may be necessary. In the third patent an autoclave is employed, a pump circulating hydrogen through the oil. When the latter has been fully treated it is drawn off to a second chamber, in which it is steamed to remove noxious gases and vapours.

The effect of hardening is to raise the melting and solidifying points, and to lower the iodine value and refractive index: the other factors are not affected, but according to W. Norman and E. Hugel the hydroxyl groups are more or less eliminated so that the acetyl value of castor oil, etc., will be affected. In the case of olein this is converted into stearine; arachidic and behenic acid are formed from marine animal oils and may be present in the hardened fats to the extent of 20 per cent., or even more; the latter acid is also formed from rape-seed oil.

The effect of hydrogenation upon various oils is shown in the table of analysis on next page.

According to an article in the *Oil, Paint, and Drug Reporter* hardened oils are now being manufactured on a very large scale both in Europe and the United States. Oils such as linseed and whale oil, being low in price, are largely employed, but not much cotton-seed oil, although the latter is now hardened to the required consistency and employed in the place of the so-called "compound lard" which consisted originally of 80 per cent. of cotton-seed oil and 20 per cent. of oleo-stearin. A little cotton-seed oil is being hardened in Europe for margarine manufacture. The total capacity of the hardening plants in Europe is estimated at 1,375,000 barrels

of 400 lb. and about half that quantity was made in 1913, while in the United States the capacity of the plant in that year was 500,000 barrels.

Hardened Oils.	Melting-Point.	Solidifying-Point.	Oleo-Refractometer at 10° C.	Acid-Number.	Saponification-Number.	Iodine-Number.	Observer.
Arachis oil			56.8	1.1	191.1	84.4	Bomer
" " (Gambia)	51.2	36.5	50.1	0.5	188.7	47.4	"
" "	44.2	30.2	52.3	1.3	188.3	56.5	"
" "	46.1	32.1	50.5	0.9	188.4	54.1	"
" "	53.5	38.8	49.0	1.2	189.0	42.2	"
" "	43.7	27.7	51.7	2.3	191.6	61.1	"
Sesame oil	47.8	33.4	51.5	0.5	190.6	51.8	"
" " (technical).	62.1	45.3	38.4	4.7	188.9	25.4	"
			at 50° C.				
Cotton-seed oil	38.5	25.4	53.8	0.6	195.7	69.7	"
" " " "	59	—	—	—	192.3	41.0	Mellana
Kapok-seed oil	55	—	39	—	191.0	32.0	"
			at 60° C.				
Soya-bean oil	68	—	42	—	190.9	15.2	"
			at 60° C.				
Coco-nut oil (natural)	25.6	20.4	37.4	0.3	255.6	11.8	Bomer
" " (hardened)	44.5	27.7	35.9	0.4	254.4	1.0	"
Whale oil	45.1	31.9	49.1	1.2	192.3	15.2	"
" "	52.2	—	29.5	—	169.5	28.8	Mellana
" "	liquid	liquid	64.1	—	192.2	144.8	Sandelin
Marine animal oil	47.2	31.9	—	1.94	189.4	24.24	Grimm
" "	38.5	31.5	—	1.00	188.3	58.34	"
Cod-liver oil	41.9	31.9	48.2	—	190.9	59.8	Sandelin
Sperm oil	50	—	—	—	141.7	17.3	"

A consular report on the trade of Marseilles in 1913 states that two factories were being built near Marseilles for hardening oils, the capital being British, and that a firm of candle makers had been hardening oils by hydrogenation for over two years. The total output of hardened oils in 1913 is put at 7000-8000 tons, which would possibly be doubled in 1914.

One firm of candle-makers were importing Japanese fish oil for treatment. Hardened oils, it is stated, fetch as high prices as copra oil, although this state of things was not likely to continue if larger quantities were produced.

With regard to detecting hardened oils in natural fats, this is a difficult problem, as the physical and many of the chemical characters are completely altered. Odour and taste entirely disappear, and the colour tests, such as Bechi or Baudoin reactions show only faintly or not at all. These hardened fats, however, contain a trace of nickel, which is readily detected by the following test: 5-10 grms. of the fat are heated in a test-tube with 10 c.c. of strong hydrochloric in a water-bath and shaken from time to time. The tube is cooled, the acid liquid separated and evaporated to dryness. The residue is then dissolved in a few drops of water and tested with a 1 per cent. solution of dimethyl-glyoxime, when if even only a minute trace of nickel be present a pink colour is produced, which is rendered more intense by addition of ammonia. The test for nickel may also be performed on the ash obtained by burning 10 grams of the oil.

CHAPTER IX.

PHYSICAL AND CHEMICAL EXAMINATION OF OILS AND FATS.

THE examination of particular oils and fats has already been dealt with under the various headings, but there are certain tests which apply to all oils and fats and these will now be considered. The tests may be divided into two groups, physical and chemical. The physical tests consist of:—

Specific gravity.

Melting-point.

Solidifying point.

Solidifying point of the fatty acids (Titre test).

Refractive index.

And the chemical tests:—

Acid number.

Ether number.

Saponification number.

Reichert-Meissl number.

Polenska number.

Hegner number.

Iodine number.

Acetyl number.

Also qualitative tests for particular oils and fats, examination of the unsaponifiable matter, and determination of glycerol when necessary.

Specific Gravity.—The specific gravity of oils is usually taken at 15° C. and that of solid fats at some temperature above the melting-point, *e.g.*, 40°, 60°, or 100° C. the com-

parison being made with water at 15° C. The determination may be carried out either with the Sprengel tube, the pyknometer, or specific gravity bottle, or the Westphal balance. The Sprengel tube consists of a U tube, both ends of which are drawn out as capillaries and bent at right angles to the limbs of the tube. There is a mark on each of the capillaries and glass caps for closing the ends. The tube is completely filled with the oil by dipping the one end into the liquid and exhausting the air by suction through the other end. The tube is then placed in a bath of water kept at 15° (40° , 60° , or 100° for fats) and after about half an hour the excess of oil is removed by placing a piece of blotting paper against one of the capillaries and slightly inclining the tube until the oil just reaches both marks on the limbs. The tube is then removed, the caps are put on, the tube carefully wiped dry and then, after standing a short time, it is weighed. The tube is also weighed empty, and filled with water at 15° . From these data, the weight of the oil and the weight of an equal volume of water are obtained from which the specific gravity is calculated.

The Westphal balance is shown in Fig. 76. This consists of a balance, one side of the beam being graduated. To this side is attached a glass rod which in this case takes the form of a thermometer for determining the temperature of the oil. There are a number of weights in the form of riders which may be hung on the hook or placed at different points on the beam as may be required. When the float is placed in water at 15° the largest weight hung on the hook will just bring about an equipoise. This equals 1.00. With oils the same rider is placed at the mark 9 on the beam and the smaller riders added until an equipoise is obtained, the total weights giving the specific gravity.

The Melting-Point.—This is determined by drawing a little of the melted fat into a capillary tube. This may be left open

or sealed off by heat. The tube is attached to the bulb of a thermometer by means of a small elastic band cut from a bit of rubber tube. The thermometer is then placed in a beaker partly filled with water, the latter being stirred while it is heated. In the open tube the fat rises as soon as it melts while in the closed tube it becomes clear. The rising-point is really the softening-point of the fat, the true melting may be

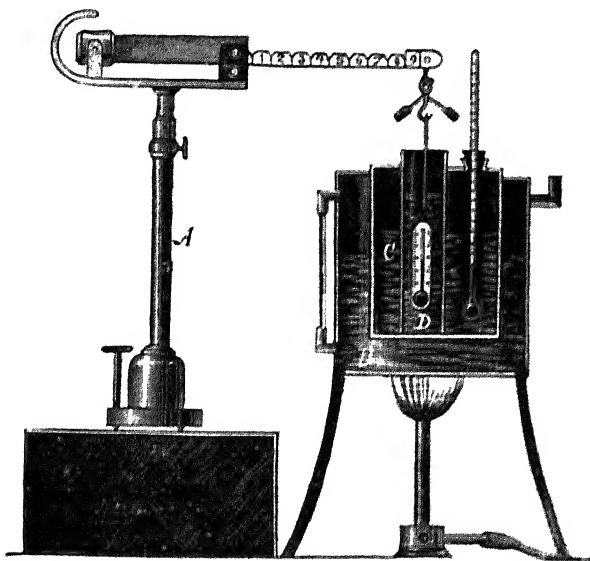


FIG. 76. Westphal balance.

a degree or two higher in some cases, on the other hand some fats become quite clear before the actual melting-point.

Solidifying and Melting-Point of Oils.—The solidifying and melting-points of oils are found by cooling them in a mixture of ice and salt, until solid, then removing and allowing to melt again, the temperature being taken by a thermometer placed in the oil.

Titre Test.—The solidifying point of the fatty acids or Titre test is an important test for fats to be used for candle-making

(see p. 171.) The fatty acids are obtained by saponifying the fat, decomposing the fat with acid, washing and drying the insoluble fatty acids and melting in a test-tube. The tube is hung in the neck of a large flask so that the fatty acids cool very slowly while they are stirred with a thermometer graduated in $\frac{1}{10}$ c.c. At the commencement of solidification there is usually a rise in temperature of 1° or 2° this point being the Titre.

Refractive Index.—This is a very useful test for determining the purity of many oils and fats, and may be carried out with only one or two drops of material. It is sometimes expressed as the true refractive index (water = 1.33) or in degrees of the Abbe butyrometer scale. The instruments used in either case are similar (see p. 111), the graduations only being different. The ordinary refractometer is the best for general purposes as it has a greater range.

Acid Number.—The acid number is the number of milligrams of potassium hydrate (KHO) required to neutralize the free fatty acids contained in one gram of a fat or oil, and is estimated by titrating a weighed quantity of the material to which alcohol has been added with $\frac{N}{10}$ potassium hydrate solution using phenolphthalein as indicator.

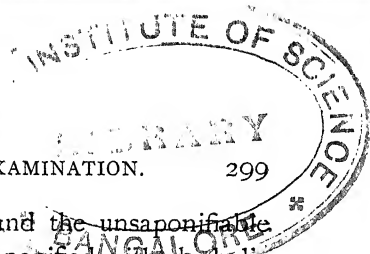
Saponification Number, which originated with Kötstorfer, and is also sometimes referred to as the "Kötstorfer number," is the number of milligrams of potassium hydrate required to saponify one gram of the fat or oil, and is determined by heating a weighed quantity of the material with a definite amount of half-normal alcoholic potassium hydrate under an inverted condenser for half an hour, the solution being then titrated with a standard solution of hydrochloric acid. A "blank" test with an equal volume of the standard solution is also performed, the difference giving the amount of alkali required for the fat or oil.

Ether Number.—The ether number is the number of milligrams of potassium hydrate required to saponify the neutral fat in one gram of the material, and is the difference between the acid and saponification numbers.

Reichert-Meissl Number.—This figure is a measure of the amount of volatile fatty acids present in the fat. It was first used by Reichert who employed $2\frac{1}{2}$ grms. of the sample. It has been more or less modified by other workers, notably Meissl and Wolny, the amount now employed being 5 grms. of material, and special methods of procedure have been laid down which it is necessary to follow in order to get concordant results. For fuller details see Lewkowitsch, *Chemical Technology and Analysis of Oils, Fats, and Waxes*, Vol. I, p. 416. Shortly, the method is as follows: 5 grms. of the fat are saponified with 2 grms. caustic potash and alcohol, the alcohol is evaporated off, the soap is decomposed with 40 c.c. dilute sulphuric acid, the liquid is distilled until 110 c.c. have passed over. The distillate is then titrated with a decinormal solution of potassium hydrate, using phenolphthalein as indicator, the number of c.c. thus used being the Reichert-Meissl number.

Polenska Number.—This figure is a measure of the insoluble volatile acids. In this method, which must also be carried out as prescribed, 5 grms. of the fat are saponified by heating with 20 grms. of glycerine and 2 c.c. of caustic soda solution (50 per cent.), the solution is diluted with 90 c.c. of water and 50 c.c. of dilute sulphuric acid (25 c.c. in 1 litre). The liquid is then distilled until 110 c.c. have passed over, the liquid is cooled to 15° C., 100 c.c. filtered and titrated as before. The insoluble acids in the flask are then rinsed on to the filter paper, dissolved in alcohol, and titrated with decinormal alkali. The number of c.c. of decinormal alkali used is the Polenska value. For further details see Lewkowitsch, *loc. cit.*, p. 424.

Hehner Number.—This figure represents the combined



amounts of the insoluble fatty acids and the unsaponifiable matter. Ten grms. of the sample are saponified with alcoholic caustic potash, the alcohol is evaporated off and the soap decomposed with slight excess of dilute sulphuric acid. The fatty acids are melted, cooled, washed with water till free from mineral acid, then dried and weighed.

Iodine Number.—The iodine value as a means of discriminating fats and oils was first employed by Hubl, whose method is described below. The solutions required for this test are:—

1. Solution of mercuric chloride in absolute alcohol, 30 grms. in 500 c.c.
2. Solution of iodine in absolute alcohol, 25 grms. in 500 c.c.

These are mixed together in equal proportions and allowed to stand 24 hours before using. This constitutes Hubl's solution.

3. Decinormal solution of sodium thiosulphate, 24.8 grms. per litre, accurately standardised against pure iodine.
4. Potassium iodide solution, 10 per cent.
5. Starch solution.
6. Pure chloroform.

To perform the test 0.2-0.3 gm. of oil or 0.5-1.0 gm. of a fat is weighed out into a clean, dry flask, 10 c.c. of chloroform is added and mixed by gentle agitation (if a fat it is melted first), 30 c.c. of Hubl's solution is then added and the flask stoppered and set aside in a dark place. A "blank" test is at the same time performed with the same quantities of the materials without the oil or fat. The time allowed for the test will depend upon the nature of the material; for most oils and fats it should be 3 hours, but drying oils require at least 18 hours. At the end of this period 15 c.c. of the potassium iodide solution is added and 40 c.c. of water, and the excess of iodine is determined with the thiosulphate solution, using a little starch paste towards the end of the operation. The

titration must be performed slowly at the last, as the violet colour does not at once disappear, and there is, therefore, a liability to overstep the mark. On standing the colourless liquid again becomes violet, but no notice must be taken of this. The "blank" test is treated in exactly the same way, the difference being due to the iodine required for the oil or fat. This is calculated on 100 grms. of the fat. For this test the pure dry filtered fat must be employed.

Hubl's test has been modified by Wij's, Haller, and Hanus, but only with regard to the solution used, the methods in all cases being essentially the same.

Wij's reagent consists of a solution of iodine monochloride in glacial acetic acid, and may be prepared by dissolving separately 7.9 grms. of iodine trichloride and 8.7 grms. of iodine in glacial acetic acid, mixing and making up to 1 litre also with glacial acetic acid. This solution retains its strength much better than Hubl's solution, and it has also a more energetic action, so that it allows of the test being completed in most cases in half an hour, and for drying oils in, at the most, 6 hours. The results are practically the same as with Hubl's solution, usually, however, rather higher and more consistent. With pure unsaturated substances it gives practically the theoretical figures. An alternative method of preparing Wij's solution is to dissolve 13 grms. of iodine in 1 litre of glacial acetic acid and pass in pure, dry chlorine gas until its titration figure is exactly doubled, which point may be noted by a marked change to a lighter colour. As soon as this occurs the current of gas should be cut off.

For dissolving the fat pure carbon tetrachloride is used in place of chloroform.

Acetyl Number.—For this test 10 grms. of the fat is heated with twice its weight of acetic anhydride under a reflux condenser. The liquid is then poured into 500 c.c. of boiling water and heated for half an hour. The acetylated fat which

separates on the surface is washed with water till free from acetic acid, and dried. Five grms. of the acetylated fat are then saponified with a known volume of alcoholic potash, the alcohol is evaporated off, the soap dissolved in water, decomposed with exactly the amount of a standard solution of sulphuric acid required to neutralize the alkali employed. The acetic acid liberated is then determined either by distillation and titration of the distillate, or by separating the solid fatty acids and titrating the filtrate. The distilled water used in all these operations must be free from carbonic acid. The acetyl number is the number of milligrams of potassium hydrate required to neutralize the acetic acid liberated from one gram of the acetylated oil or fat.

Unsaponifiable Matter.—This will contain cholesterin (and also phytosterin if vegetable fats are present). If paraffin wax is present this will also be found in the unsaponifiable matter; for the method of detection see p. 151. Mineral oils and rosin oils may also be present as adulterants. Unsaponifiable hydrocarbons have, however, been detected as normal constituents in some of the shark oils, see p. 23.

Cholesterol is present in all animal fats and oils; on the other hand, all vegetable fats and oils contain an isomeric substance named phytosterol or sitosterol. The sterols can be isolated from the unsaponifiable matter by recrystallising several times from alcohol. The appearance of the crystals is characteristic, but they are best identified by their melting-points, that of cholesterol being 148° - 150° C, and that of sitosterol, 136° - 137° C. The sterols can be converted into their acetates by heating with acetic anhydride, and the acetates, after recrystallising several times from absolute alcohol, show very definite melting-points, pure cholesterol acetate melting at 114° C. and sitosterol acetate, 127° C.

In some cases it is very important to know, as, for instance, with margarine, whether a fat is a pure animal or a pure

vegetable fat or a mixture of the two; in such a case this test is the most conclusive.

Recently the sterols have been isolated from fats and oils by means of digitonin. According to Klostermann and Opitz the material must first be saponified and the unsaponifiable matter separated in the usual way, but Berz and Augerhansen state that it is sufficient to add an alcoholic solution of digitonin direct to the fat or oil, and, after adding chloroform, separate the alcohol, which contains the digitonide in suspension, the latter being then filtered off. This is confirmed by Marcusson and Schilling, who work in a similar manner. The compounds formed by the action of digitonin on the sterols are known as digitonides; by heating with acetic acid they are converted into the corresponding acetates, which after purification by recrystallisation as above described can be examined in the usual manner.

The following colour tests for cholesterol may also be applied to the unsaponifiable portion:—

Hager's Test as Modified by Salkowsky.—The material to be tested should be dissolved in about 2 c.c. of chloroform, about 2 c.c. of strong sulphuric acid is now added and the mixture shaken. If cholesterol is present the liquid assumes a blood-red colour which changes to a cherry-red and then purple.

Liebermann's Test.—The material is dissolved in acetic acid and a little strong sulphuric acid is poured carefully down the side of the tube. In presence of cholesterol a violet-coloured ring is formed.

Note.—Rosin, rosin acids, and rosin oils give somewhat similar reactions, so that if any of these are present the tests would not be reliable.

The following general observations on the physical and chemical constants may be found useful:—

Melting-Point.—This increases with the percentage of solid

glycerides and especially with the stearin. The solidifying point is usually 10° or 12° below the melting-point. The melting and solidifying points of the fatty acids are usually slightly higher than those of the fats and are close to each other.

Specific Gravity.—The specific gravities of some of the blubber oils are extremely low, *e.g.*, sperm oil ($0.876-0.883$), some animal oils moderately low, *e.g.*, neatsfoot oil, horse oil ($0.913-0.920$). The gravities of most fats and oils at 15° C. lie between 0.920 and 0.935 .

Refractive Index.—This constant increases directly with the specific gravity in most instances, an exception is sperm oil, which with a low gravity has a moderately high refractive index.

Acid Number.—This is usually low in animal fats, but varies with the quality of the raw material and the length of storage.

Saponification Number.—This is low with sperm oil ($128-132$) and high in butter fat ($220-233$). With most fats and oils it varies between 190 and 198 . This depends upon the molecular weights of the glycerides or other compounds present.

Reichert-Meissl Number.—This gives a measure of the amount of volatile fatty acids (butyric, caproic, caprylic). Most fats contain only traces of volatile fatty acids, equal to $\cdot 3$ to $\cdot 5$ per cent. of butyric acid, butter contains 6 to 8 per cent., coco-nut fat about 4 per cent., and palm-nut fat about 3 per cent.

Hehner Number.—This represents the amount of insoluble fatty acids plus the unsaponifiable matter. Usually the latter does not exceed $0.3-0.5$ per cent., but in sperm oil it averages about 40 per cent. The Hehner number of most oils and fats is about 95.5 , that of butter fat $86-88$, coco-nut fat $88-90$, and palm-nut fat 91 .

Iodine Number.—This varies with the amount of unsaturated glycerides (linolin, linolenin, etc.) present. Thus it is low with solid fats and high with oils, especially drying oils and fish oils.

The nature of the oil or fat can be determined by separating the fatty acids having lead salts soluble in ether and estimating their iodine value. The following are the iodine values of these unsaturated acids:—

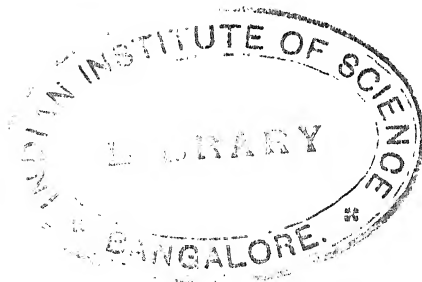
Name.	Formula.	Atoms of I for 1 Molecule of Acid.	Iodine Number, Per Cent
Hypogaeic. . . .	$C_{16}H_{30}O_2$	2	100.0
Physetoleic . . .	$C_{16}H_{30}O_2$	2	100.0
Oleic	$C_{18}H_{34}O_2$	2	91.07
Jecoleic	$C_{18}H_{32}O_2$	2	85.81
Brucic	$C_{22}H_{42}O_2$	2	75.15
Ricinoleic . . .	$C_{18}H_{34}O_3$	2	85.24
Linolic	$C_{18}H_{32}O_2$	4	181.43
Linolenic . . .	$C_{18}H_{30}O_2$	6	274.10
Jecoric	$C_{18}H_{30}O_2$	6	274.10
Clupanodonic . .	$C_{14}H_{26}O_2$	8	108.11

Acetyl Number. This is a measure of the hydroxylated fatty acids contained in an oil. The figures, except in a few cases, are small, varying from 1.0 in coco-nut fat to 2.2 in horse fat, according to Lewkowitsch.

In the examination of fats it is not alone a question of identifying a certain fat and establishing its purity, but regard must also be paid to the presence of soaps, free fatty acids and extraneous non-fatty matters, such as water, resins, and hydrocarbons. Soaps may be removed by shaking up the carbon bisulphide solution of the fat with water. The presence of free fatty acids in fats, except in large amount, cannot be considered an adulteration, since they are generally present in small amount. Occasionally, however, the occurrence of free fatty acids indicates decomposition (rancidity) of the fat. In the absence of free fatty acids, rosin can be detected by shaking up the oil with moderately strong alcohol and evaporating

the latter. The separation of rosin acids from free fatty acids is most suitably effected by the method prescribed by T. S. Gladding, which is based on the ready solubility of silver resinate in ether, whereas silver nitrate, oleate, stearate, etc., are almost completely insoluble therein. Also by Twitchell's process, the fatty acids being dissolved in alcohol and dry hydrochloric acid passed in whereby the true fatty acids are converted into esters, which are neutral, while the rosin acids are left unaltered and, after washing to remove the mineral acid, can be estimated by titration. Hydrocarbons can, in general, be detected by saponifying the sample with alcoholic potash (5 grms. of oil, 2 grms. of potassium hydroxide, 25 c.c. of 90 per cent. alcohol), the resulting soap being mixed with pure sand, the alcohol driven off by evaporation on the water-bath at a temperature not exceeding 50° C., and the residue extracted with ether or petroleum spirit, the hydrocarbons present being left behind on the evaporation of the solution.

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